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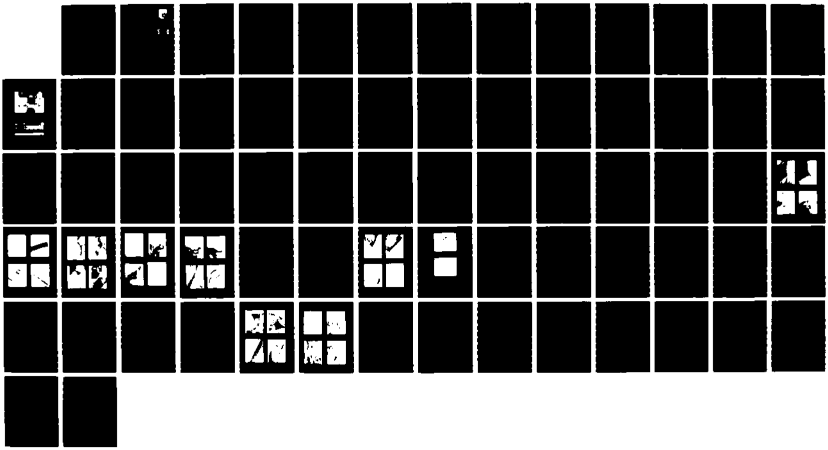

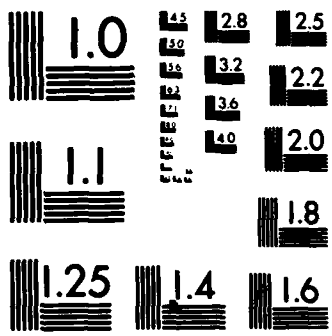

MICROCOPY RESOLUTION TEST CHART
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CARBON RESIDUE STUDIES WITH A MICRO CARBON RESIDUE TESTER

Wilhelm Bochartz

Lubrication Branch
Fuels and Lubrication Division

June 1986

Final Report for Period November 1984 - October 1985

Approved for public release; distribution unlimited

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This technical report has been reviewed and is approved for publication.

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A test procedure for the coking propensity of gas turbine lubricants was developed using the ™Micro Carbon Residue Tester-100™ (MCRI-100). The MCRT-100, a microprocessor controlled heating unit, was evaluated for its ability to determine carbon residue in weight percent of synthetic gas turbine lubricants under controlled static conditions (various time/temperature profiles and selected gas atmospheres). The purpose was to yield information on the amount of deposit remaining in glass vials after a measured volume of lubricant had been exposed to different degrading environments, varying the parameters of temperature, gas (air or nitrogen), and exposure time. During a test, several processes occur simultaneously. The major effect is volatilization of the lubricant. Substantial oxidation and thermal degradation of the lubricant also occurs, which forms residue in the glass vials. The degradation of the lubricant is not completely realistic because the evaporated oil is caught in the condensate trap of the MCRT-100, whereas condensate returns

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19. ABSTRACT (Cont'd)

to the bulk oil in an engine and affects viscosity, acidity, and the degradation of the lubricant.

The residue after complete evaporation consists mainly of deposits from the breakdown of the lubricant with traces of additives. However, if the temperature is very high (330°C and higher) the deposits undergo further oxidation to form volatile products and the final deposits are significantly lower on a weight basis.

Criteria for evaluating the test lubricants is the weight of the remaining vial deposits. The basic studies were conducted with four 3 cSt lubricants meeting MIL-L-7808J specification and two 5 cSt lubricants meeting MIL-L-23699C specification. Tests at different temperature (340°C to 500°C) at times up to 15 hours showed that these test temperatures were too high because the lubricants evaporated before coking occurred. Degradation with nitrogen compared to air was very low. The actual temperature of the lubricant sample was 10-14% higher than the displayed temperature on the MCRT. The temperatures in different positions in the MCRT did not vary significantly.

Repeatability at different times/temperatures was excellent. Time/temperature profiles at 250°C, 275°C, 300°C, and 330°C up to 96 hours show that the 3 cSt oils have approximately 50% less residue compared to the 5 cSt oils. One possible explanation for this is the higher evaporation loss of the 3 cSt oils. The time/temperature profiles also show that, for practical test purposes, 275°C for 30 hours or 300°C for 15 hours are the best test conditions.

Tests conducted at different airflow rates (150 and 600 cc/min at 20 psig and 1000 cc/min at 34 psig) showed that an increased airflow rate (150 cc/min and 600 cc/min) results in an increased amount of residue. However, the amount of residue decreased slightly when airflow was increased to 1000 cc/min compared to 150 cc/min. It also could be seen that the repeatability was reduced at higher airflow rates versus a lower rate of 150 cc/min.

Scanning electron microscope (SEM) studies and carbon-hydrogen-nitrogen (C-H-N) analysis were used to compare residues and indicates that direct comparisons between residue from the MCRT, the J57 engine and the J57 engine simulator are difficult. It is believed that the different conditions in an engine (static and dynamic processes and many other parameters which influence the formation of deposits) and the MCRT (only static process, no oil/metal contact) is the reason for the differences observed.

A test procedure for the coking propensity of synthetic gas turbine was established and used on 14 different oil samples.

Lubricant 0-82-3 produced the least amount of deposit with an average of 8.09 $\pm$ 0,24 wt, 0-82-2 a middle range average deposit of 17.70 $\pm$ 0.20 wt% and TEL-5028 the highest amount of deposit with an average of 27.78 $\pm$ 0.36 wt% at 275°C for 30 hours.

The evaluation also shows that the MCRT-100 has potential use as a cost effective laboratory device to differentiate lubricants regarding their tendencies to form coke deposits prior to engine test. However, the MCRT-100 simulates only one condition in an engine (static) and cannot replace deposition testers which simulate dynamic processes.

PREFACE

This technical report was prepared by the Lubrication Branch, Fuels and Lubrication Division, Aero Propulsion Laboratory (APL), Air Force Wright Aeronautical Laboratories (AFWAL), Air Force Systems Command, Wright-Patterson Air Force Base, Ohio. The work herein was accomplished under Project 3048, Task 304806, Work Units 30480618 and 30480626, "Turbine Engine Lubricant Development" and "Turbine Engine Lubricant Research", during the period of November 1984 to October 1985 with Mr W. R. Bochartz as Project Engineer. Special acknowledgement is given to Mr P. W. Centers and Mr G. A. Beane IV, AFWAL/POSL, for their outstanding contribution to this effort.

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SECTION I

INTRODUCTION

Current aircraft turbine engine lubricants (3 or 5 cSt) are qualified regarding many physical, chemical and performance properties according to the specifications MIL-L-7808J or MIL-L-23699C, respectively. One of these performance properties is "Deposition Tendency". Such evaluations are performed since formation of deposits in oil systems of turbine engines can be a significant problem. The deposits reduce the heat transfer of lubricant heat exchangers and, after deposits break loose from engine surfaces, they may plug lubricant jets and filters, hinder the operation of bearings and seals, and degrade the performance of other engine components. Aside from the prime consideration of possible in-flight malfunction, engine deposits also significantly increase the time and cost of maintenance.

In the near future, lubricant deposits in gas turbine engines will be of more concern using current 3 and 5 cSt oils because higher engine pressure ratios and increased turbine temperatures will produce an increase in the thermal and oxidative stress on lubricants.

As a consequence, considerable effort has been devoted to the development of suitable bench or rig tests which could be used in characterizing lubricant deposit performance prior to engine tests.

There are often four steps in the evaluation of a synthetic gas turbine lubricant before it is qualified for use in engines: initial laboratory tests, bench testing, engine simulator testing, and full scale engine testing. Although the final qualification of a lubricant depends on its performance in a full-scale engine test, it is first tested with less costly test procedures. Laboratory and bench tests can provide useful data at low cost, because it is obvious that the earlier in the qualification process an oil can be disqualified for use in an engine the less costly the evaluation process will be.

All the following deposition related tests may be used in achieving this goal, but only numbers 3 and 10 are used in the MIL-L-7808J specification and numbers 3, 10 and 13 are used before a new oil candidate is tested in the J57-P29 full scale

engine test. Numbers 4 and 10 are used in the MIL-L-23699C specification and numbers 4, 10 and 15 are used before a new oil candidate will be tested in a T-63 engine.

1. RTD panel coker.
2. Oxidation-corrosion-deposition (O-C-D) test.
3. WADC deposition test.
4. High-temperature deposition test.
5. Thin film oxidation test (TFD).
6. Static coker.
7. Inclined panel coker.
8. Incline plane coker.
9. Rotating cylinder.
10. Bearing deposition test.
11. Hot-wall deposition test.
12. RTD seal rig.
13. Engine simulator.
14. Differential thermal analysis and thermogravimetric analysis.
15. Vapor phase coker.

There is still no single test device and many turbine engine manufacturers, oil manufacturers, and lubricant qualification laboratories use their own in-house developed deposition test procedures.

To make the qualification of lubricants cost-effective, it would be advantageous to have a worldwide accepted standardized test device and test procedure for the determination and comparison of the coking propensity of synthetic gas turbine lubricants suitable for use in universal lubricant specification.

The "Micro Carbon Residue Tester - (MCRT-100)", developed by Alcor Incorporated, San Antonio, Texas is a microprocessor controlled heating unit originally designed to duplicate the results of the corrosion carbon test (ASTM-D-189). The test may be performed under various time/temperature profiles and selected oven atmospheres (air or nitrogen). Thus, the need to evaluate the MCRT-100 since it may provide a basis for such a standardized test device.

SECTION II

EXPERIMENTAL APPARATUS AND PROCEDURE

1. EXPERIMENTAL APPARATUS

The Micro Carbon Residue Tester (MCRT-100) is a microprocessor controlled heating unit designed to assess carbon residue from lubricants under various time/temperature profiles and selected oven atmospheres (air or nitrogen). The test apparatus is shown in Figure 1. A schematic of the oven and the gas flow system is shown in Figure 2.

The temperature in the oven may be increased from ambient to 600°C at heating rates from less than 1°C up to 50°C per minute. During the test, the oven is continuously purged with air or nitrogen, which sweeps the evolved vapor from the oven, while maintaining a controlled atmosphere in the heating chamber. The oil/gas vapor flows downward and is retained in the condensate trap or exits through the exhaust chimney. Gas flow through the oven is fixed by internal needle valves which permit delivery of 150 cc/min or 600 cc/min at 20 psig on the MCRT gauge.

The temperature of the oven is measured by a thermocouple which protrudes through the oven wall (Figure 2). The outer oven wall is insulated by a thick mantle to hold the temperature in the oven constant during the tests. After placing the sample holder in the oven, it is closed with a gravity seal lid.

If, during a test, the gas pressure drops from 20 psig to about 3 psig or the power goes off, then the controller goes into "remote hold" or shows "flag" and the test stops so that the operator is aware that there was a gas or power failure.

Test sequences are controlled by the microprocessor, which can store a maximum of 12 programs with 8 segments per program. Each segment in a program must be programmed for the "end set point" of the segment the "time" of the segment and the "status" of the four event markers (either on or off). Figure 3 shows an example program (tests at 340°C/15 hours) with segment times, temperatures, and events.

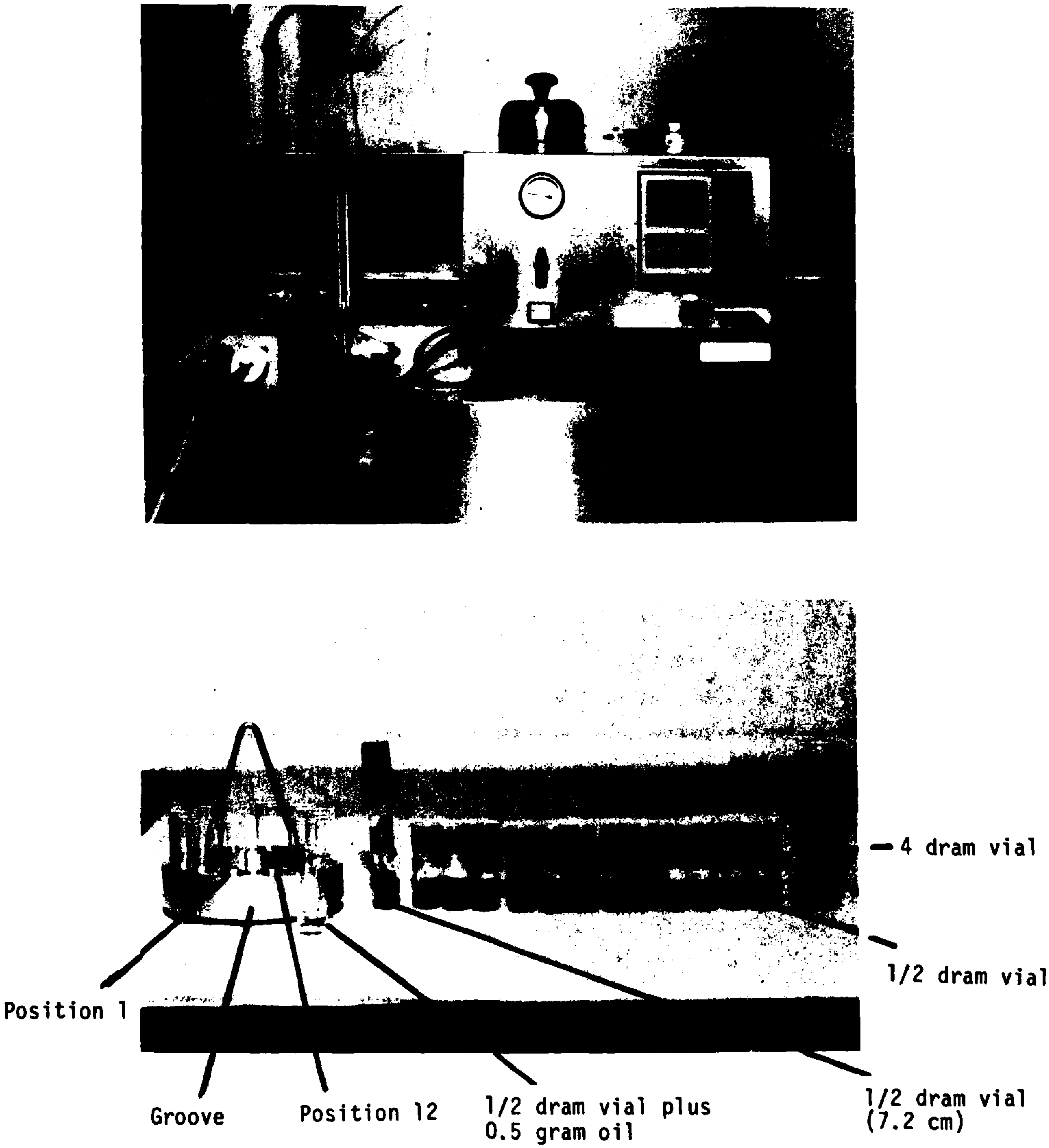


Figure 1. Micro-carbon Residue Tester Apparatus and Aluminum Sample Holder Showing Position Numbers

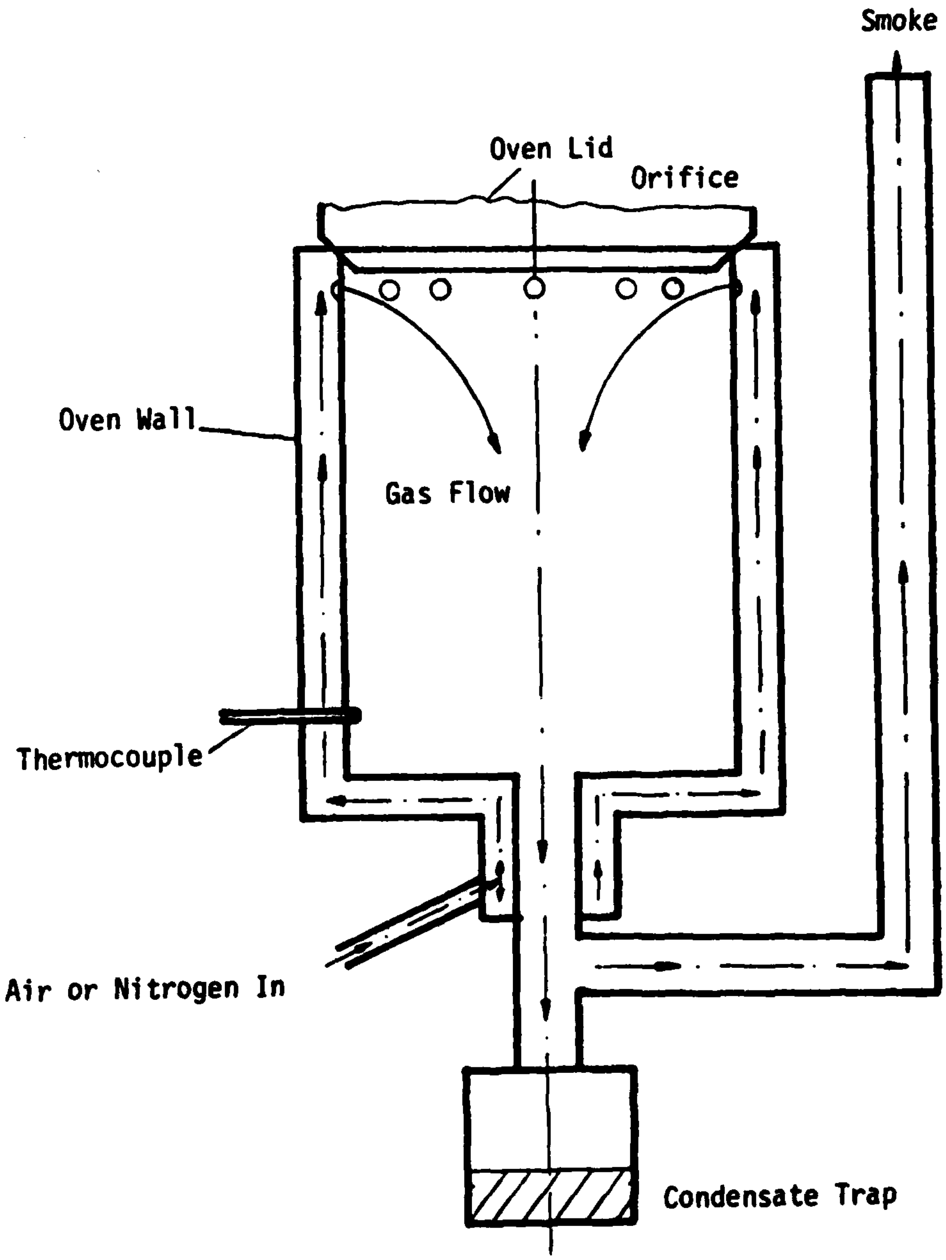
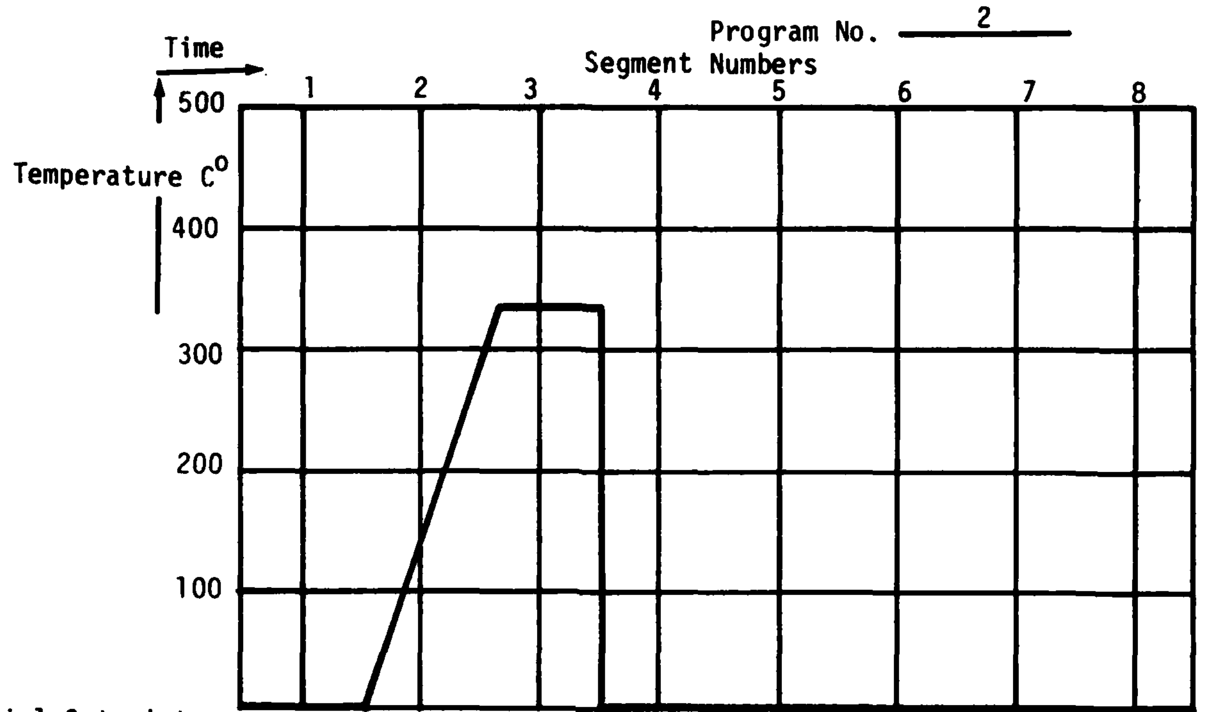


Figure 2. Schematic of the MCRT-100 Oven and the Gas Flow System



Initial Setpoint & 8 End Points									
	0	0	340	340	0	0	0	0	0

Segment Time		0	0.5hrs	15 hrs	0	0	0	0	0
Events	1	ON	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Mark Events	2	OFF	OFF	OFF	OFF	OFF	OFF	OFF	OFF
Either ON	3	OFF	ON	OFF	OFF	OFF	OFF	OFF	OFF
or OFF	4	OFF	ON	ON	ON	ON	ON	ON	ON

The control tuning constants and the events are:

Cycle time = 2
Out/limit = 50
Prop band = 10
Reset = 1
Rate = 0.1
Load line = 15

EVENTS - These are program aids and controlled functions as follows:
EVENT 1 - For use in zero time first step of any program as an indication that a program has been reset due to power failure/restore. This causes REM to light.
EVENT 2 - Used to turn on automatic reset inhibition* when making an abrupt increase in setpoint, so overshoot is minimized.
EVENT 3 - Turns on additional gas flow for purge and cooling times.
EVENT 4 - Recommended for use to indicate program is running (same as RUN) but flashes via a closed loop when program is completed.

*i.e., it turns off the integral action control mode.

Figure 3. Example Program for Tests Conducted at 340°C for 15 Hours Test Time

Any or all programs can be linked to form up to a 96 segment program. Each segment time can be specified as either 0 to 99 minutes or 0 to 99 hours.

2. EXPERIMENTAL PROCEDURE

The operation procedure used for conducting the tests is presented below.

a. The first step in the procedure is to clean the glass vials. Handle cleaned vials only with forceps, because any sample or other material on the vials affects the accuracy significantly.

b. Weigh and record weight of the cleaned vials (1 to 12 per test).

c. Add 0.5 gram test lubricant in each of the vials (weigh to the nearest 0.1 mg) and record the weight.

d. Put the filled vials in order 1 to 12 into the sample holder (Figure 1.) which is arranged so that the groove on the basket lies between 1 and 12 on an imaginary clock.

e. Lower the sample holder into the oven so that the groove of the aluminum basket is placed in line with the thermocouple that protrudes through the oven wall and put the lid on the oven.

f. Turn on the power and the air supply (air at 20 psi on the MCRT gauge).

g. Program the microprocessor (initial set point, segment time/temperature events and control tuning constants).

h. Start the test by pushing the nitrogen switch on the MCRT.

i. Allow the oven to cool to room temperature after the test.

j. Remove and weigh the samples.

k. Record data and calculate percent residue.

TABLE 1

EXAMPLE OF DATA METHOD

Vial numbers (identical with positions on sample holder)	1	2	... 12
Weight of vials plus sample (Start)	2.7343	2.7032	2.7522
Weight of vials	2.2342	2.2032	2.2522
Weight of oil sample	0.5001	0.5000	0.5000
Weight of vial plus residue (End)	2.3044	2.2705	2.3245
Weight of vial	2.2342	2.2032	2.2522
Weight of residue	0.0702	0.0673	0.0723
Residue % = $\frac{\text{Weight of residue}}{\text{Weight of oil sample}}$ X 100%	14.04	13.46	14.46

SECTION III

LUBRICANTS EVALUATED

A total of 14 lubricants were evaluated at different times/temperatures. Table 2 presents a listing of lubricants used in this program. Results of viscosity at 40°C, total acid number (TAN), and evaporation loss (after 6.5 hours at 400°F) tests, as well as specification data are given for these lubricants.

TABLE 2

LUBRICANTS EVALUATED

OIL CODE	VISCOSITY AT 40°C (cSt)	TAN mg KOH/g	EVAPORATION LOSS WT %	DESCRIPTION
0-71-6	25.74	0.03	3.0	MIL-L-23699C
0-72-11*	12.4	0.24	25	MIL-L-7808J
0-75-1B/S**	--	--	--	
0-77-15	23.90	0.49	3.1	MIL-L-23699C
0-78-8	13.84	0.20	16	MIL-L-7808J
0-78-9	13.06	0.22	20	"
0-79-16	12.4	0.21	25	"
0-79-17	13.5	0.04	21	"
0.82-2	12.8	0.05	14	MIL-L-7808H
0.82-3	14.2	0.06	25	MIL-L-7808J
0.82-14	13.6	0.11	18	MIL-L-7808 Type
TEL-5027	26.58	0.58	3.6	D.ENG.RD 2497
TEL5028	26.13	0.55	3.6	"
TEL-5029	22.86	0.05	3.7	"

*0-72-11 = 0-79-16 but different batch
**0-75-1 B/S = Basestock from 0-79-17

SECTION IV

TEST RESULTS AND DISCUSSIONS

The following report summarizes the progress made on the evaluation of the MCRT-100 capabilities.

1. SELECTION OF PROPER TEST TEMPERATURE, TIME AND SAMPLE WEIGHT

The preliminary evaluation of the MCRT-100 was initiated using the proposed test procedure from the manufacturer to see how much residue could be expected at 500°C, 14 minutes test time and different sample weights (1.0 and 2.0 gram). The results obtained indicated that a chamber temperature of 500°C is too high for these lubricants due to excessive volatilization. It was, therefore, decided to use a temperature range from 250°C to 380°C and different times up to 96 hours. This time/temperature profile was chosen because it is believed that the deposits in the J57 engine and the J57 engine simulator are formed at those temperatures during a 100-hour test. Tests at 340°C and 380°C and different times from 2 to 15 hours (test program, see Figure 3), a changed ramp time/temperature (0.5 hours to 340°C or 380°C), different sample weights (0.5, 1.0 and 2.0 gram), and 150 cc/min nitrogen flow showed that the amount of residue remaining in the glass vials was still very low (0.63± 0.03% residue was the maximum result). The test data showed also that there was a loss of lubricant from the 1/2 dram vials when using 1.0 and 2.0 gram lubricant samples, and so it was decided that for the next tests only 0.5 gram oil per vial or less should be used at a temperature from 250°C to 330°C and a test time from 5 to 96 hours.

The data also indicated that the degradation of the lubricant sample with pure nitrogen was not significant, so subsequent tests were conducted with air instead of nitrogen.

2. ACTUAL TEMPERATURE OF LUBRICANT SAMPLE VERSUS THE DISPLAY TEMPERATURE ON THE MCRT-100

Before additional tests were performed, the variation in lubricant sample temperature in different positions in the MCRT-100 oven were tested by threading a Type-J (Iron-Constantan) thermocouple through the exhaust chimney and submerging it in a 0.5 gram sample of MIL-L-7808 lubricant. The actual lubricant temperature was then compared to indicated MCRT temperatures of 250, 275, 300 and 330°C. The indicated temperature on the MCRT was measured from the installed thermocouple, which protrudes through the oven wall 1.5 centimeters above the bottom of the oven. Data presented in Table 3 indicate that although the sample temperatures were independent of vial position the actual sample temperatures were significantly higher than indicated on the MCRT display.

TABLE 3

TEMPERATURE OF LUBRICANT SAMPLE IN DIFFERENT POSITIONS OF THE MCRT OVEN COMPARED TO THE DISPLAY TEMPERATURE ON THE MCRT (MCRT set temperature = 275°C, 12 vials, 0.5 gram lubricant each, airflow 150 cc/min).

POSITION OF VIALS	ACTUAL TEMPERATURE OF SAMPLE °C*	DISPLAY TEMPERATURE/°C
1	287 ± 0.5°C	275°C
2	286 " "	"
3	" " "	"
4	287 " "	"
5	" " "	"
6	288 " "	"
7	286 " "	"
8	287 " "	"
9	286 " "	"
10	287 " "	"
11	286 " "	"
12	287 " "	"

*A check on the accuracy of the thermocouple (Type-J) was determined by immersion in an ice point bath.

Figure 4 shows that the actual lubricant sample temperature in position six is 13°C higher than the display temperature. It also shows that the temperature in the oven during a test is very stable (±0.5°C) after the set temperature (275°C) or the actual lubricant sample temperature (288 ± 0.5°C) was reached.

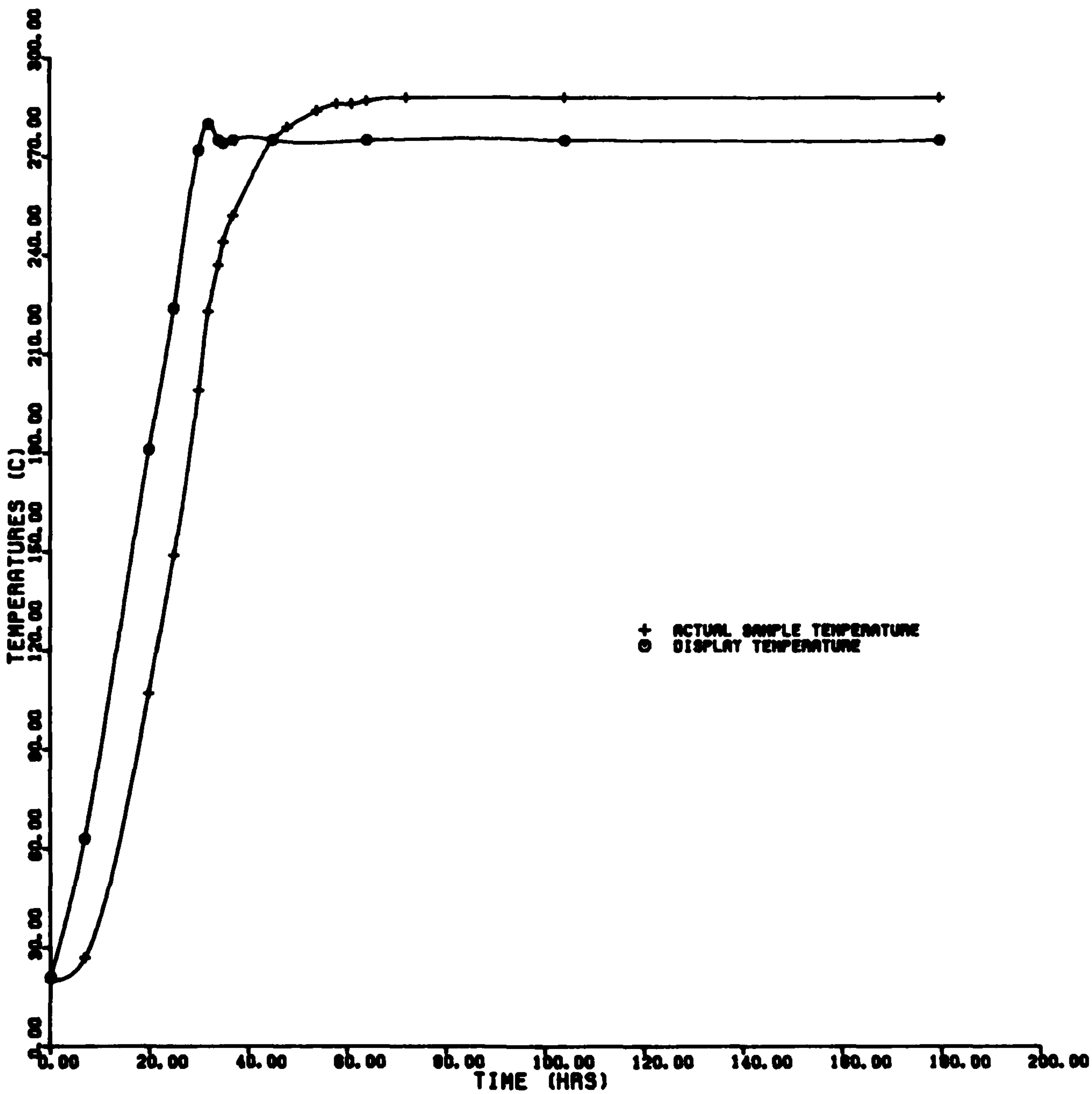


FIGURE 4. ACTUAL VS DISPLAY TEMPERATURE PROFILE OF LUBRICANT SAMPLE IN POSITION 6 AT 275 C

Figure 4. Actual Versus Display Temperature Profile of Lubricant Sample in Position Six at 275°C

Table 4 gives the actual temperature of the lubricant sample in position six compared to the MCRT set temperature of 250, 275, 300, and 330°C. The measurements show that the actual temperature of lubricant sample in the MCRT-100 oven were higher by about 10-14°C than indicated and it was found that the sample temperature in different positions around the MCRT oven is sufficiently consistent. The investigation also indicated that the temperature in the oven during a test is very stable (±0.5°C) after the set temperature is reached. The actual sample temperature variance in the MCRT oven was not considered a significant problem for subsequent evaluation, and therefore, it was decided to use the temperature as displayed on the MCRT.

TABLE 4

ACTUAL TEMPERATURE OF LUBRICANT SAMPLE IN POSITION SIX COMPARED TO SET TEMPERATURE

SET TEMPERATURE (°C)	SAMPLE TEMPERATURE (°C)
250	260 ± 0.5
275	288 ± 0.5
300	314 ± 0.5
330	344 ± 0.5

TABLE 5

DATA OF ACTUAL LUBRICANT SAMPLE TEMPERATURE (°C) IN THE MCRT OVEN FROM TWO DIFFERENT TEST UNITS AT 275°C

POSITION OF VIALS	UNIT 1 (ROLLS ROYCE-DATA)	AERO PROPULSION UNIT 2 (POSL-DATA)
1	286	287
4	289	287
6	285	288
7	285	286
10	288	287

3. REPEATABILITY TESTS WITH THE PROPOSED TEST PROCEDURE FOR 5 cSt OILS FROM ROLLS ROYCE (UK)

a. Rolls Royce (UK) had already done some testing with the MCRT and proposed the following test procedure: 1/2 hour heating period to 150°C (high purge on), 1 hour at 150°C to remove all light ends from the oils, 1/2 hour heating period to 275°C (high purge on) and then 30 hours at 275°C. The repeatability tests at 275°C for 30 hours test time with four lubricants meeting MIL-L-7808J specification (0-72-11, 0-79-17) and meeting MIL-L-23699C specification (0-71-6), 0-77-15) are given in Table 6. The test program is shown in Table 6a.

TABLE 6

REPEATABILITY TESTS AT 275°C FOR 30 HOURS WITH FOUR LUBRICANTS

	RESIDUE (WT%)			
	OIL CODES			
TEST NO.	0-71-6	0-77-15	0-79-17	0-72-11
1	22.37 ± 0.16	23.99 ± 0.57	9.60 ± 0.40	13.52 ± 0.56
2	22.79 ± 0.14	23.83 ± 0.35	9.66 ± 0.42	14.06 ± 0.54
3	22.76 ± 0.29	23.67 ± 0.33	9.93 ± 0.39	14.12 ± 0.30
4	--	--	9.74 ± 0.42	12.92 ± 0.44
Mean and Standard deviation	22.64 ± 0.23	23.83 ± 0.16	9.73 ± 0.14	13.66 ± 0.56

TABLE 6a

PROPOSED TEST PROGRAM FOR FIVE cSt LUBRICANTS FROM ROLLS ROYCE

SEGMENT	INITIAL SET POINT TEMP./°C	END POINT TEMP./°C	TIME/HRS	EVENTS 1	2	3	4
2-1	0	0	0	ON	OFF	OFF	OFF
-2	--	150	0.5	OFF	ON	ON	ON
-3	--	150	1.0	OFF	OFF	OFF	ON
-4	--	275	0.5	OFF	ON	ON	ON
-5	--	275	30.0	OFF	OFF	OFF	ON
-6	--	0	0	OFF	OFF	ON	ON
-7	--	0	0	OFF	OFF	OFF	ON
-8	--	0	0	OFF	OFF	OFF	ON
3-1	--	0	0	OFF	OFF	OFF	OFF

TABLE 7

COMPARISON OF REPEATABILITY DATA FROM TWO DIFFERENT TEST UNITS
WITH LUBRICANT 0-71-6 (AT 275°C FOR 30 HRS)

	RESIDUE (WT%)	
TEST NUMBER	UNIT 1 (ROLLS ROYCE)	UNIT 2 (AERO PROPULSION)
1	21.55 ± 0.39	22.37 ± 0.16
2	22.21 ± 0.62	22.79 ± 0.14
3	22.25 ± 0.43	22.76 ± 0.29
Mean of Three Tests:	22.00 ± 0.39	22.64 ± 0.23

The data from Tables 6 and 7 indicate excellent repeatability from test to test (Table 6) and a good repeatability between two different test units (Table 7). However, additional data will be required before an overall test repeatability statement can be made.

b. Figure 5 shows the actual lubricant sample temperature in position 1 to 12 in the MCRT-100 oven and the remaining residue in wt% from lubricant 0-77-15 and 0-72-11 at 275°C for 30 hours in these positions.

It can be seen that the amount of residue differs slightly between position number 1 to 12. The data also indicate that the 3 cSt oils have approximately 50% less residue compared to the 5 cSt oils at the same test conditions. One possible explanation for the difference in the amount of remaining residue is the higher evaporation loss of the 3 cSt lubricants compared to the 5 cSt lubricants under the specified test conditions. The 3 cSt lubricants show an evaporation loss from 16% to 25%, whereas the 5 cSt lubricants show an evaporation loss from 3 to 3.7% at the same test conditions (6 1/2 hours test time at 400°F; Table 2).

4. REPEATABILITY TESTS WITH FOUR LUBRICANTS AT A TIME COMPARED TO ONE LUBRICANT AT A TIME TESTED IN THE MCRT OVEN

The data from Table 6 indicate excellent repeatability, when using 12 vials and one lubricant at a time in the MCRT oven.

The next tests were conducted to see if it is possible to use more than one lubricant at a time in the MCRT to reduce the test time and still have the same repeatability.

TABLE 8

COMPARISON OF REPEATABILITY TESTS WITH FOUR LUBRICANTS AT A TIME VERSUS ONE LUBRICANT AT A TIME IN THE MCRT OVEN.

	RESIDUE (WT%)*			
	OIL CODE			
Tests With	0-71-6*	0-77-15*	0-72-11**	0-79-17**
One oil at a time in the oven	22.64 ± 0.23	23.84 ± 0.16	13.59 ± 0.60	9.88 ± 0.42
Four oils at a time in the oven	21.88 ± 0.17	24.14 ± 0.43	13.56 ± 0.43	9.84 ± 0.14

* Means of 3 tests.
** Means of 5 tests.

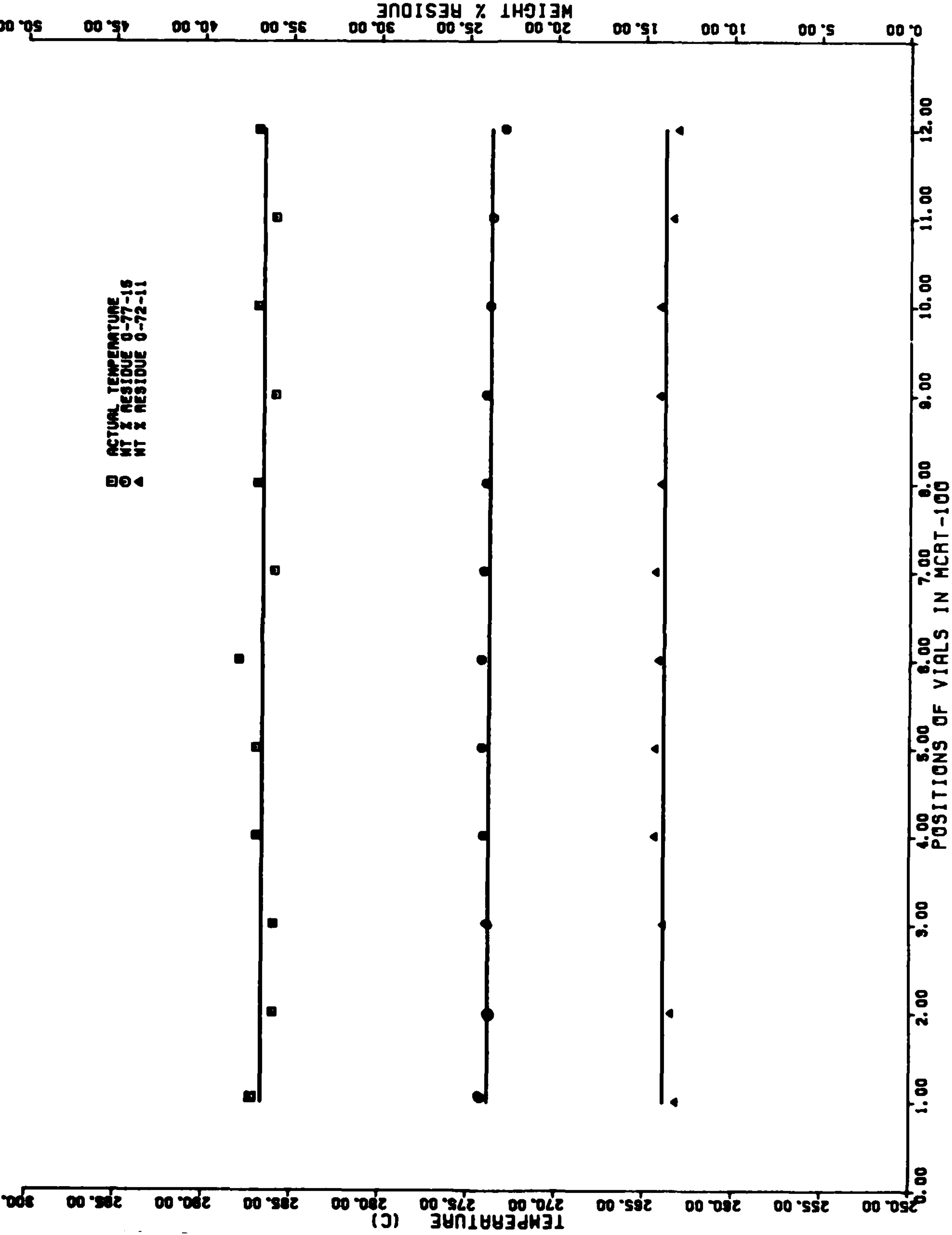


Figure 5. Actual Lubricant Sample Temperature and Weight Percent Residue in Vial Positions 1 to 12 in MCRT-100

The comparison of the data from Table 8 indicate even with four lubricants evaluated concurrently that repeatability is excellent. Therefore, it was decided that the following tests would be conducted with four lubricants concurrently in the MCRT oven.

The positions of the vials for the tests with four lubricants in the oven were the following:

(a) For the first test:

Lubricant 0-71-6 position of vials 1, 5 and 9
" 0-77-15 " " " 2, 6 and 10
" 0-72-11 " " " 3, 7 and 11
" 0-79-17 " " " 4, 8 and 12

(b) For the second test: rotate the lubricant positions.

Lubricant 0-79-17 was placed at positions 1, 5 and 9
" 0-71-6 was placed in positions 2, 6 and 10
" 0-77-15 " " " " 3, 7 and 11
" 0-72-11 " " " " 4, 8 and 12

(c) For the third test:

Lubricant 0-72-11 positions of the vials 1, 5 and 9
" 0-79-17 " " " " 2, 6 and 10
Lubricant 0-77-15 positions of the vials 3, 7 and 11
" 0-71-6 " " " " 4, 8 and 12

The rotation of the vials from test to test shall be conducted as described previously. The reason for the rotation of the vials is to get the best possible repeatability, because in some vial positions the remaining amount of residue is always slightly higher.

5. COMPARISON OF DATA FROM TESTS WITH DIFFERENT OVEN ATMOSPHERES (AIR AND NITROGEN)

TABLE 9

COMPARISON OF DATA FROM TESTS WITH 150 CC/MIN NITROGEN FLOW VERSUS 150 CC/MIN AIRFLOW AT 275°C FOR 30 HOURS

	RESIDUE (WT%)	
OIL CODE	150 CC/MIN NITROGEN FLOW	150 CC/MIN AIRFLOW
0-72-11	5.66 ± 1.91	13.59 ± 0.60
0-79-17	3.94 ± 1.51	9.88 ± 0.42

The above data indicates that the remaining residue with airflow is between 57% (0-72-11) and 60% (0-79-17) higher compared to the results with nitrogen flow. That indicates that the degradation with pure nitrogen is very low. The poor repeatability with nitrogen cannot be explained. However, all subsequent tests were carried out with airflow instead of nitrogen flow.

6. TEMPERATURE PROFILES OF FOUR LUBRICANTS AT DIFFERENT TEMPERATURES (250°C TO 330°C) UP TO 96 HOURS TEST TIME AND 150 CC/MIN AIRFLOW

Several tests were carried out at 250, 275, 300, and 330°C for up to 96 hours with four lubricants. (Two lubricants meeting MIL-L-23699C specification (0-71-6, 0-77-15) and two lubricants meeting MIL-L-7808J specification (0-72-11 and 0-79-17). Test conditions were the same as for the repeatability tests. The mean and standard deviation of the remaining residue were calculated and plotted against the test time. Figures 6, 7, 8, and 9 give residue as a function of time of four lubricants at 250, 275, 300, and 330°C. Figures 10, 11, 12, and 13 give the residue as a function of time from each individual lubricant.

It can be seen (Figure 6) that the time required until only solid residue remains in the glass vials at a temperature of 250°C is too long for practical test purposes. The residue versus time curve flattened out after 50 hours test time with lubricant 0-72-11 and 0-79-17 (3 cSt) and was still decreasing after 96 hours with lubricant 0-71-6 and 0-77-15 (5 cSt).

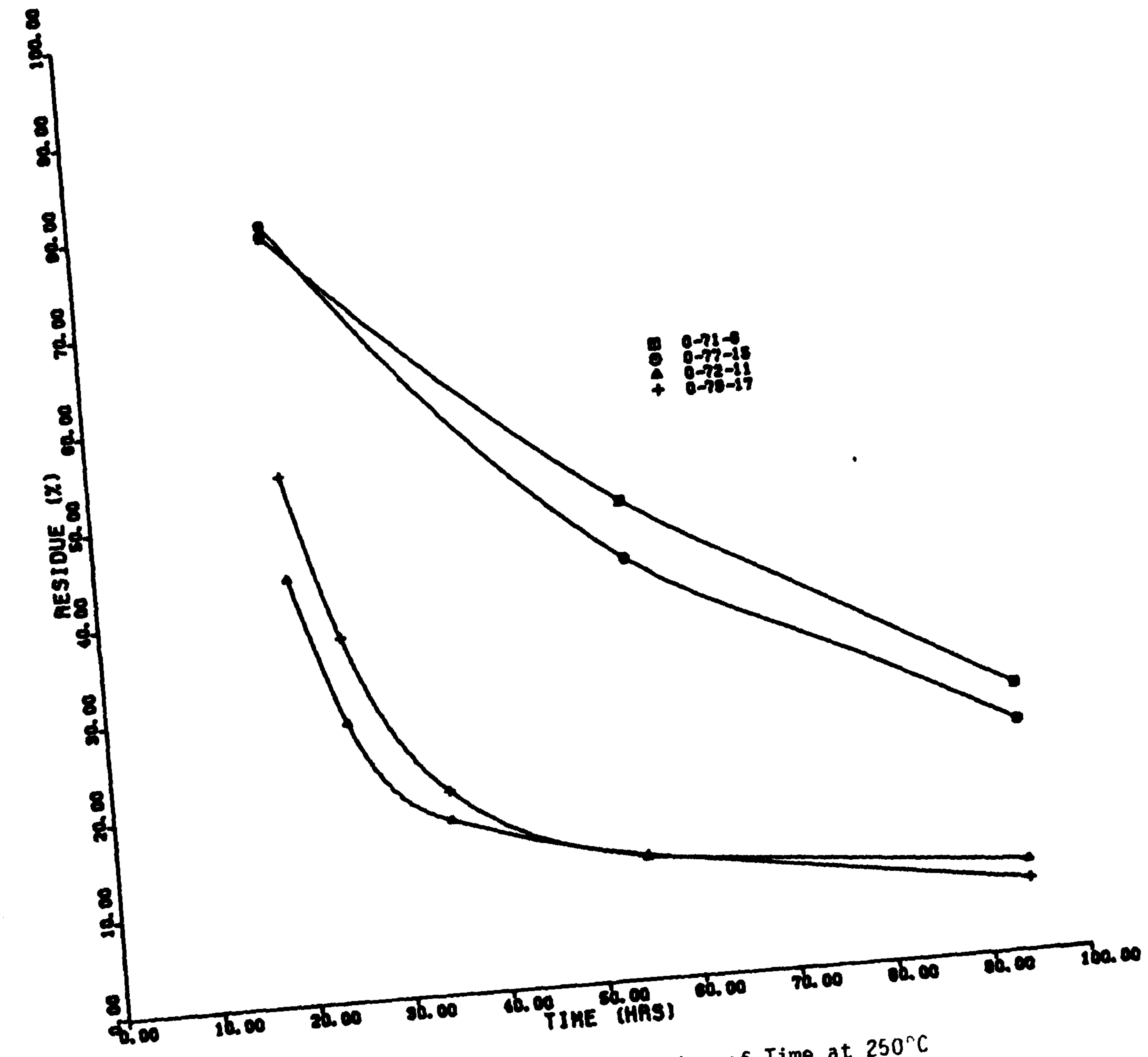


Figure 6. Residue as a Function of Time at 250°C

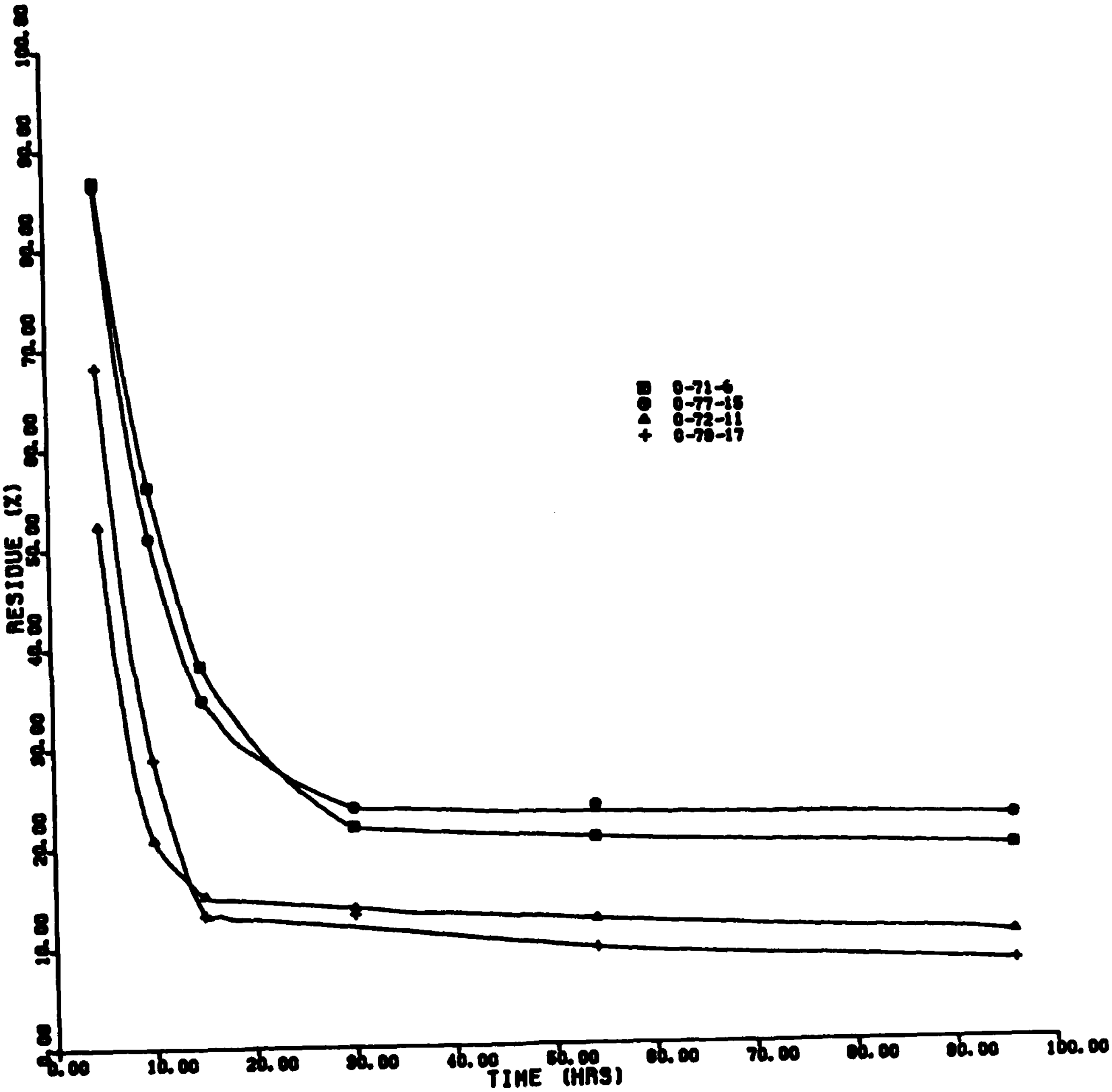


Figure 7. Residue as a Function of Time at 275°C

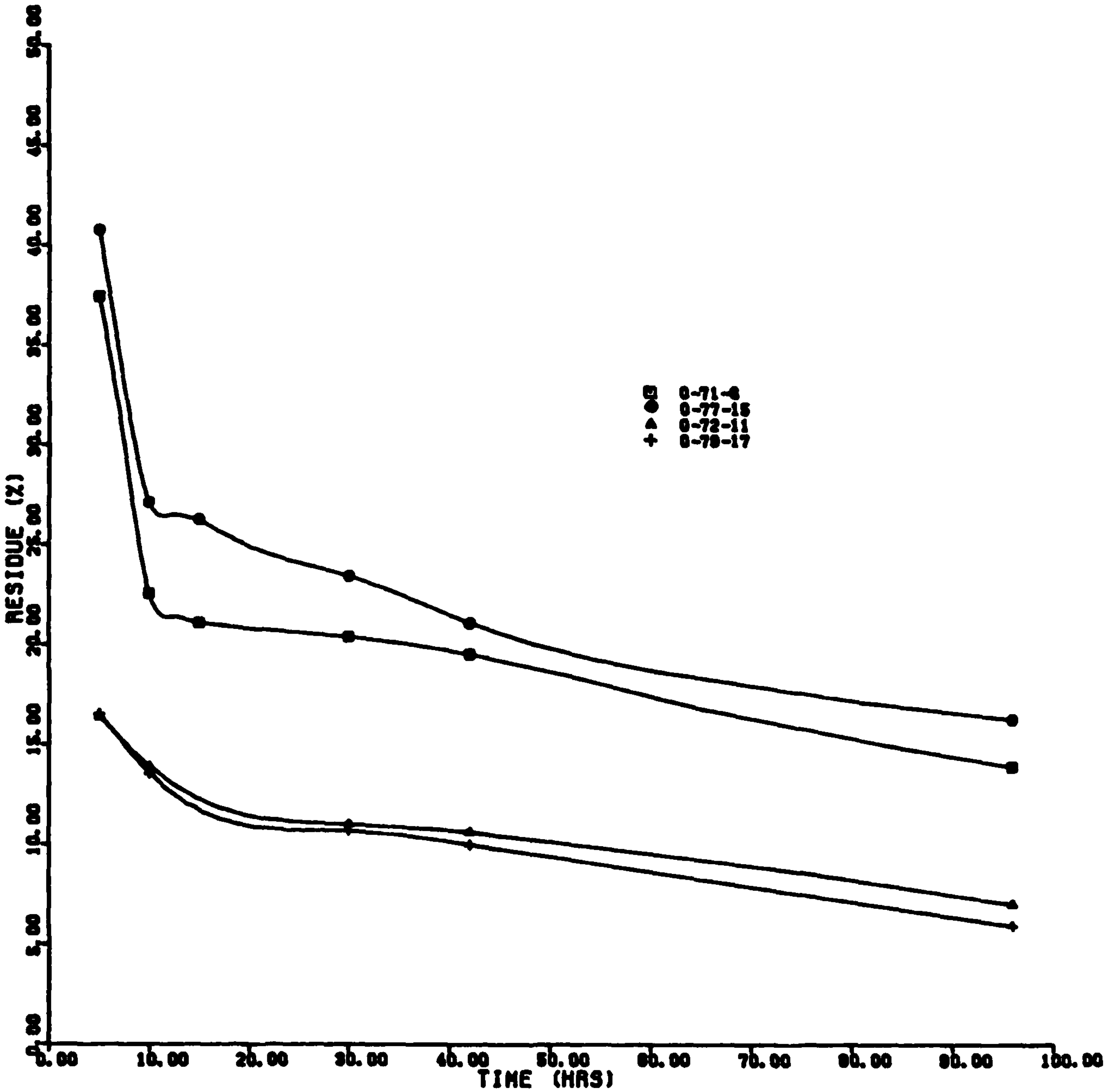


Figure 8. Residue as a Function of Time at 300°C

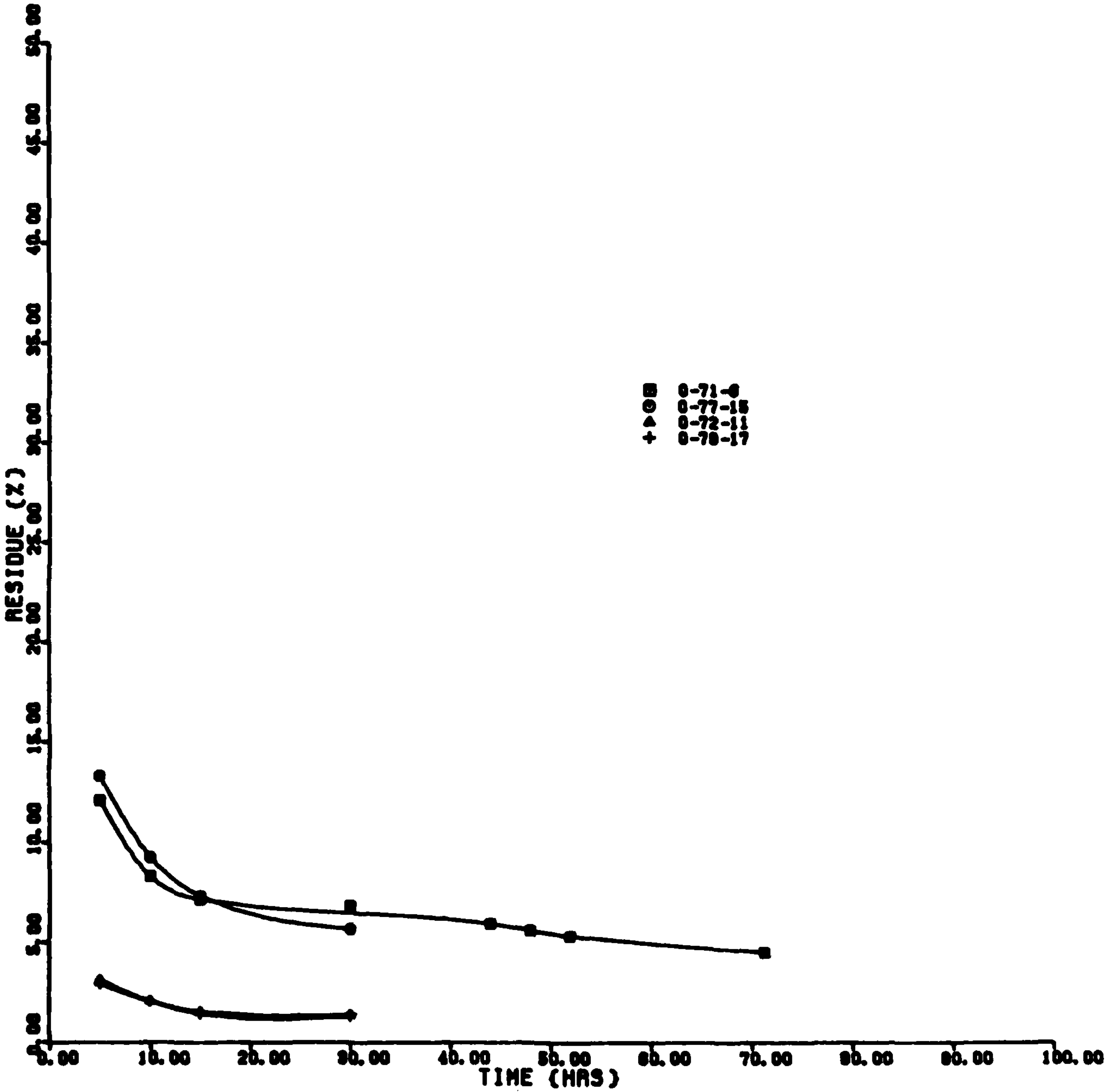


Figure 9. Residue as a Function of Time at 330°C

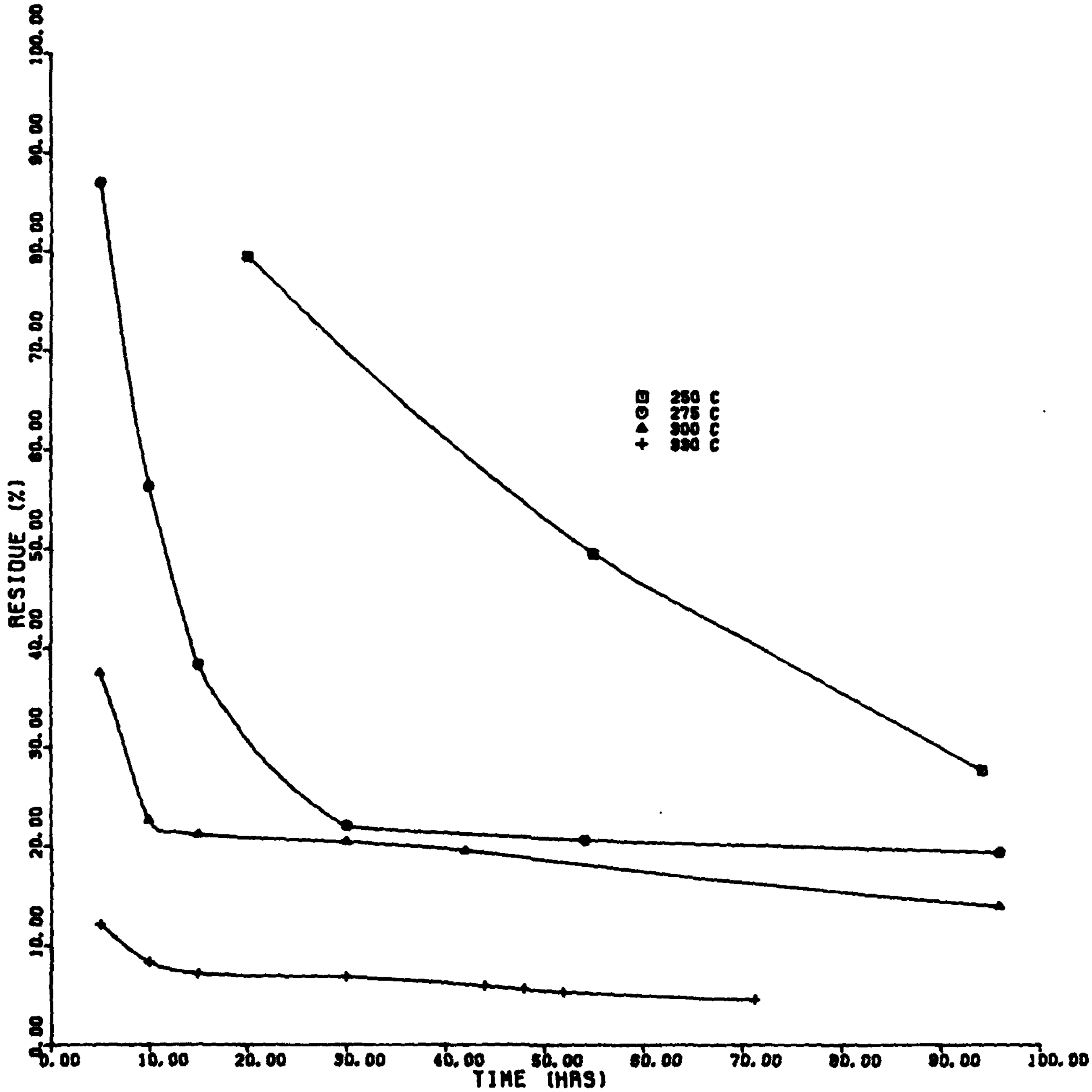


Figure 10. Residue as a Function of Time (0-71-6) at 250, 275, 300, and 330°C

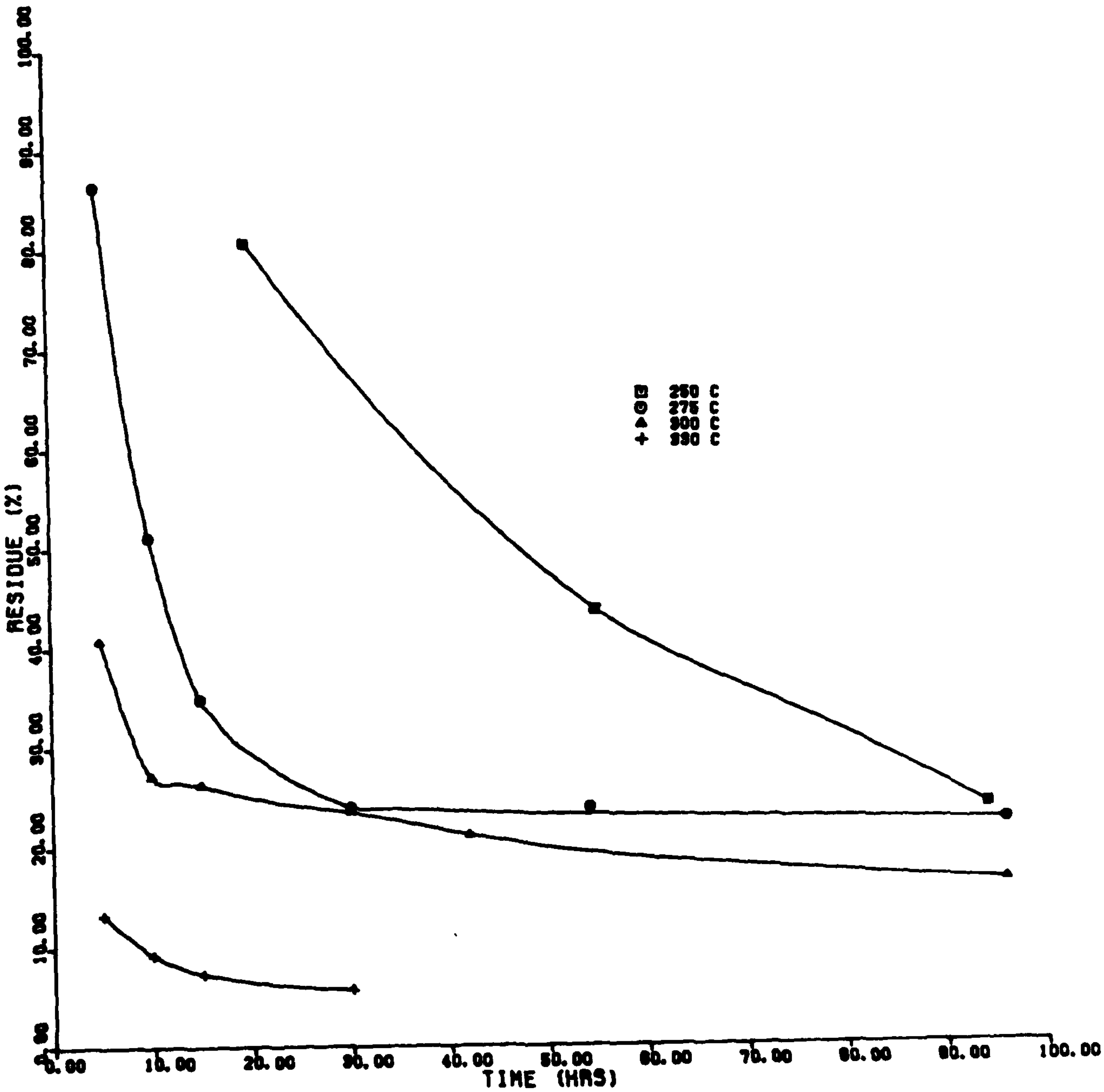


Figure 11. Residue as a Function of Time (0-77-15) at 250, 275, 300, and 330°C

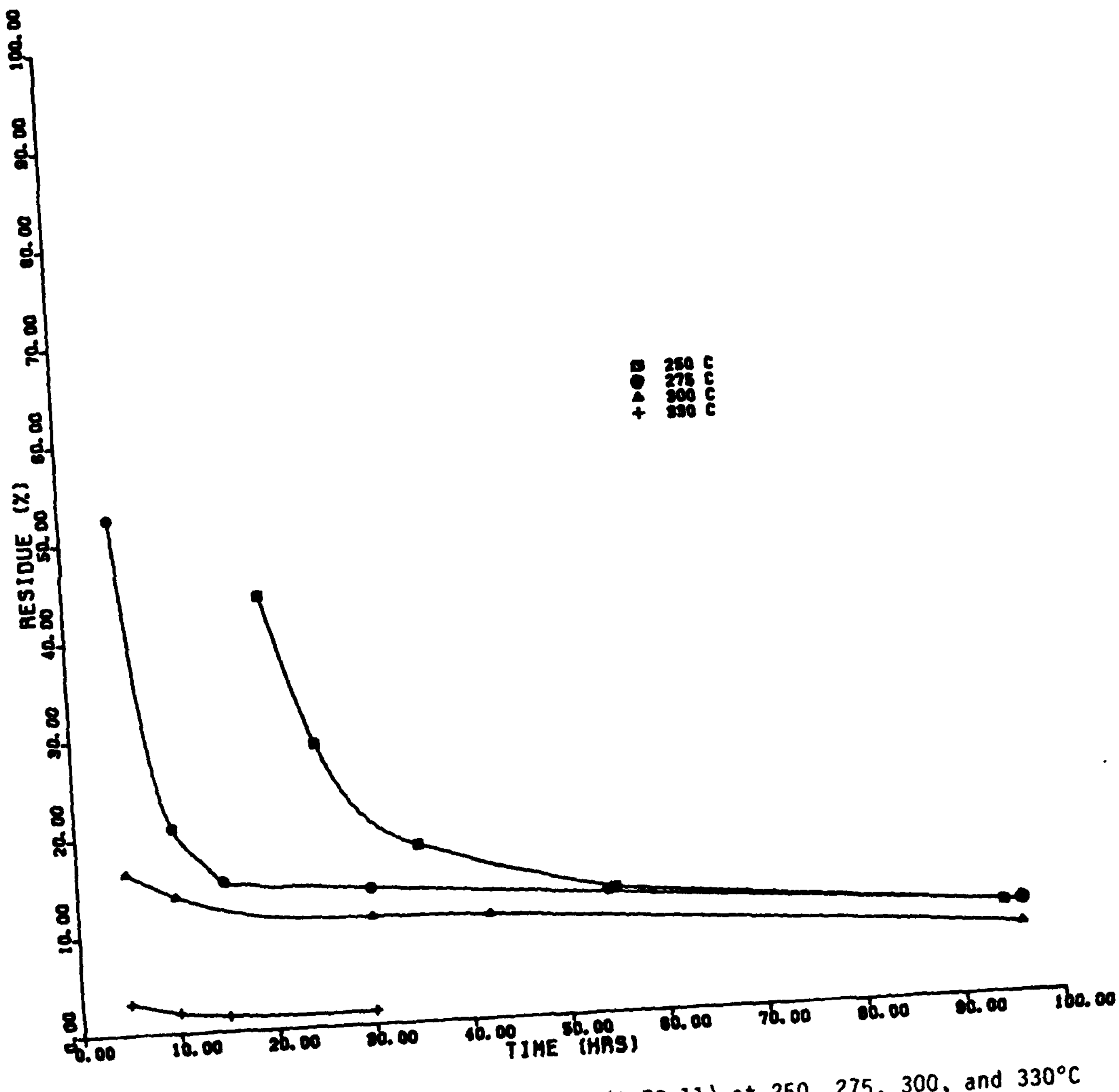


Figure 12. Residue as a Function of Time (0-72-11) at 250, 275, 300, and 330°C

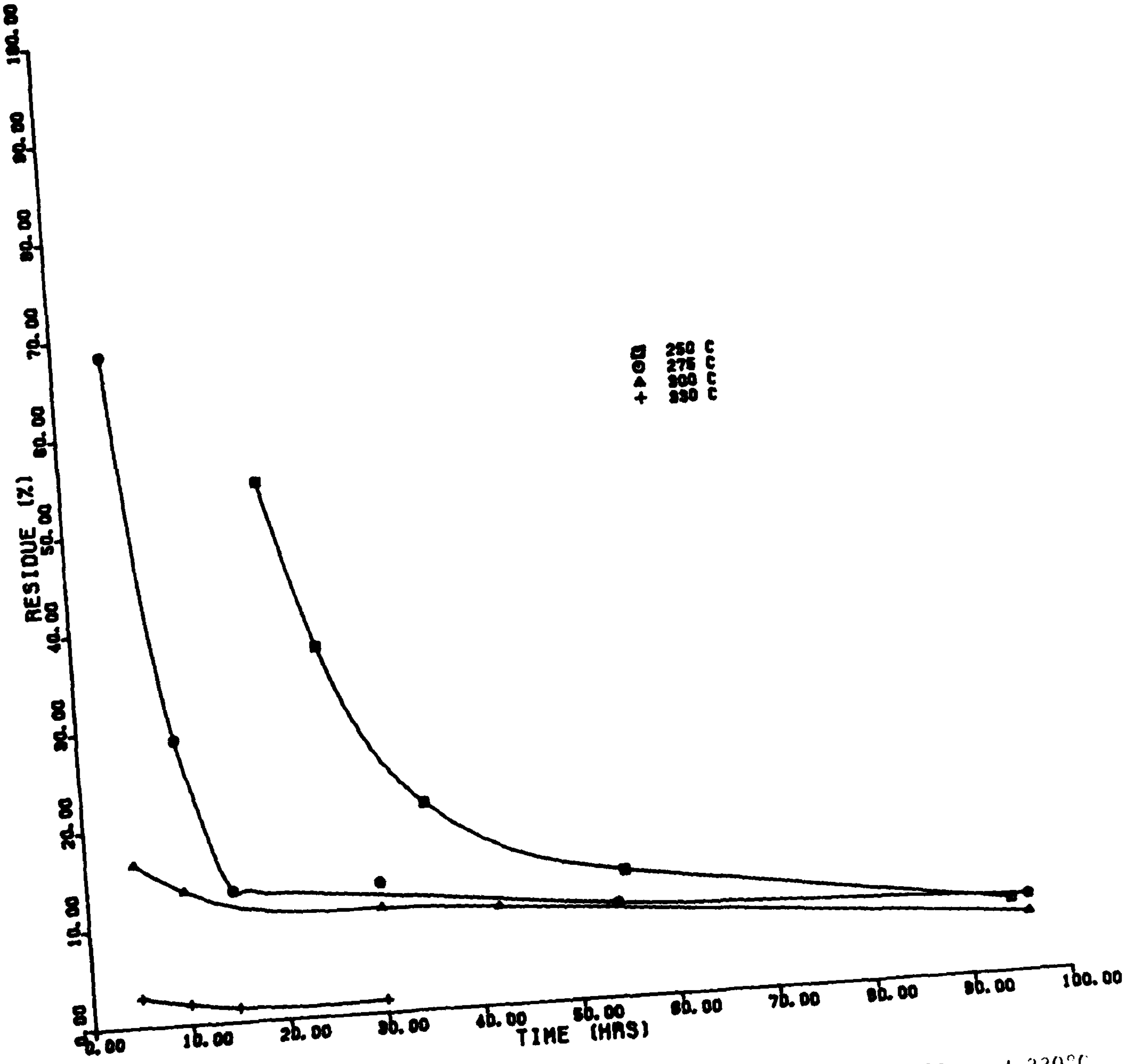


Figure 13. Residue as a Function of Time (0-79-17) at 250, 275, 300, and 330°C

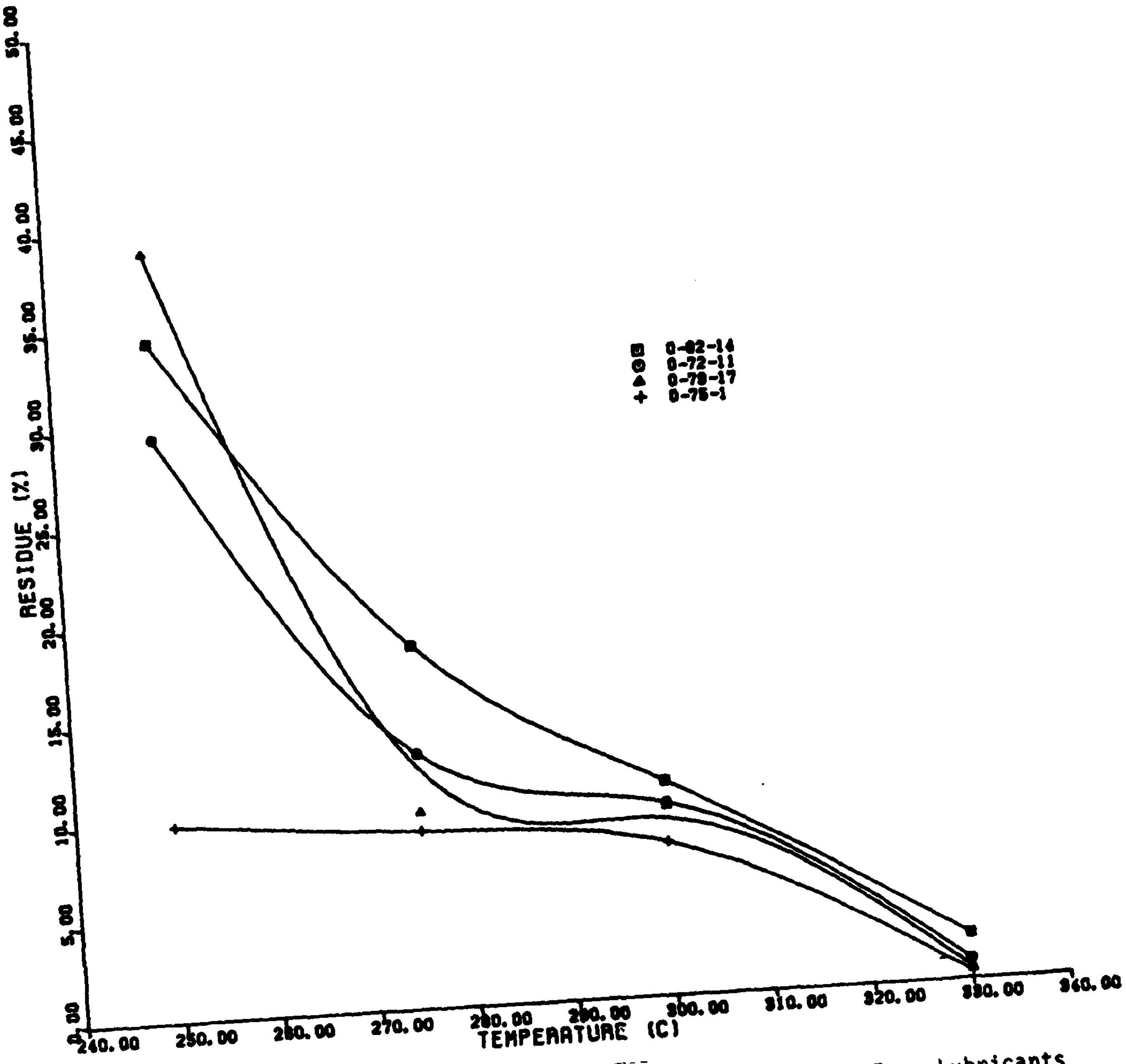


Figure 14. Residue as a Function of Test Temperature with Four Lubricants
Test Time 30 Hours

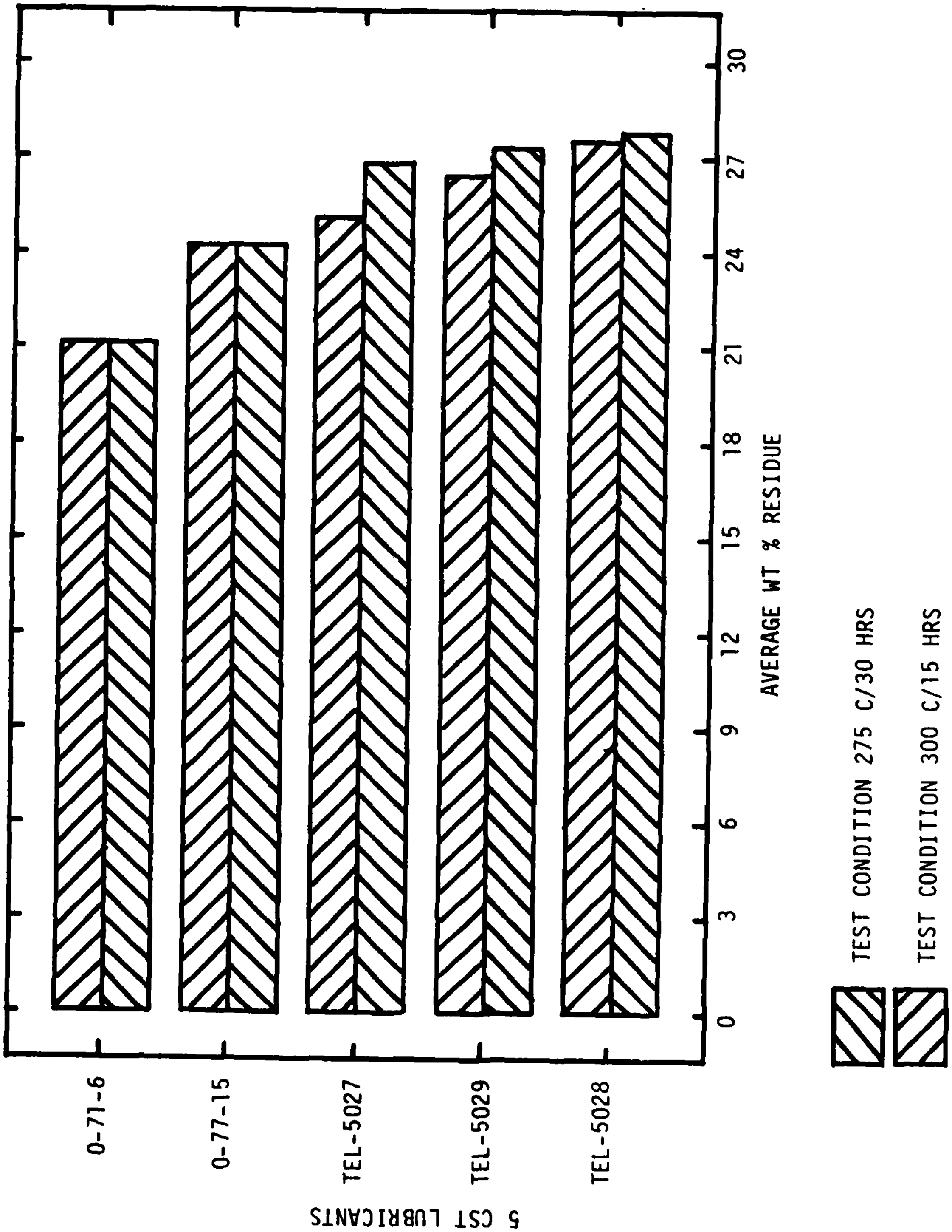


Figure 15. Residue MCRT-100 at 275°C for 30 Hours and 300°C for 15 Hours (5 cSt Lubricants)

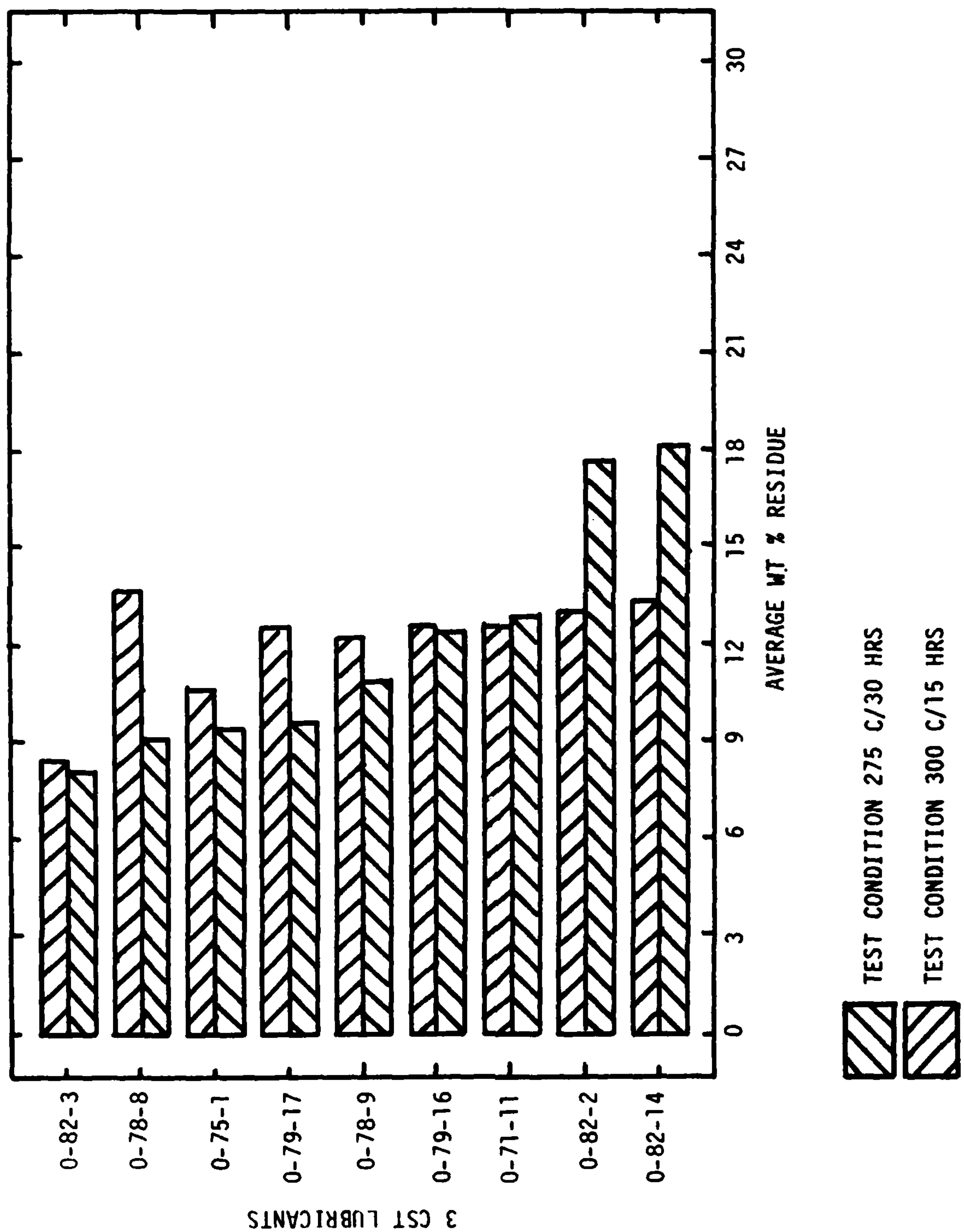


Figure 16. Residue MCRT-100 at 275°C for 30 Hours and 300°C for 15 Hours (3 cSt Lubricants)

The time/temperature profiles (Figure 9) indicate that at 330°C and five hours test time, about 88% (5 cSt) and 97% (3 cSt) of the lubricants were evaporated and that the solid residue after 30 hours test time was very low. The results presented indicate that 330°C (which is actually 344°C) is too high, because much lubricant evaporated after five-hour test time and the remaining deposits underwent further oxidation and formed volatile products which is the reason why the amount of solid residue was not stable and continued to decrease with time. On the other hand, there was nearly no difference in the amount of residue with lubricants from the same specification so that a differentiation was not possible. For that reason, no further tests at 330°C were conducted.

At 275°C (Figure 7), the residue versus time curve flattened out after 20 hours test time with the 3 cSt lubricants and flattened out after 30 hours with the 5 cSt lubricants. At 300°C (Figure 8), the residue versus time curve flattened out after 15-hour test time, but the decrease of the amount of residue after 96 hours at 300°C was slightly higher than at 275°C. Based on all the data, it appears that the best test time and temperature for practical test purposes are 275°C and 30 hours test time or 300°C and 15 hours test time.

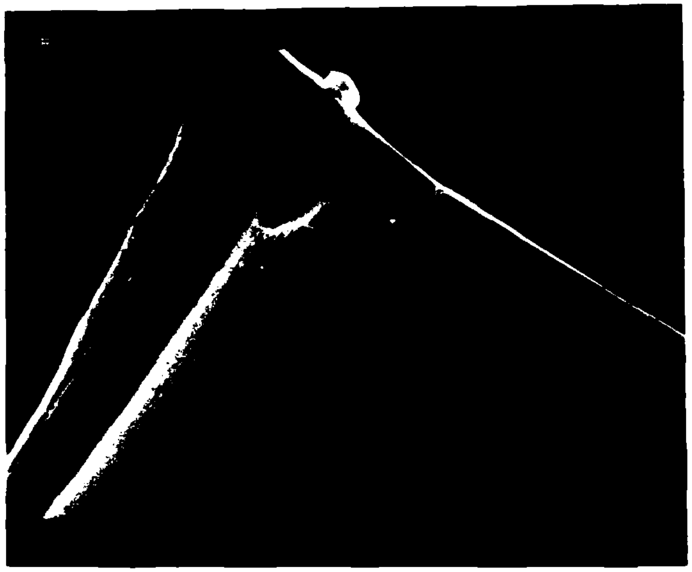
Figure 17 shows SEM pictures of lubricant 0-79-17 residue obtained from the MCRT at 275°C after 30 hours and 300°C after 15 hours. The pictures indicate no difference in the morphology at the different test conditions. Table 19 presents C-H-N analyses of 0-79-17 residue from the MCRT at the above test conditions and indicate that only a slight difference in the C-H-N content could be observed. The content at 300°C after 15 hours is 4.61 wt% (C) and 0.23 wt% (N) higher than the C and N content at 275°C after 30 hours whereas the H content is 0.41 wt% lower at 300°C after 15 hours. Figures 17 and 18 are SEM pictures of 0-79-17 residue obtained from the MCRT-100 at 275°C after 30 hours and different test samples (#8, 11 and 24). It can be seen that there was no change in the morphology of the residue with the same oil (0-79-17) and the same test conditions, but different tests. The temperature profiles at 250, 275, 300, and 330°C up to 96 hours test time show that the best test time and temperature for practical test purposes are 275°C for 30 hours or 300°C for 15 hours.

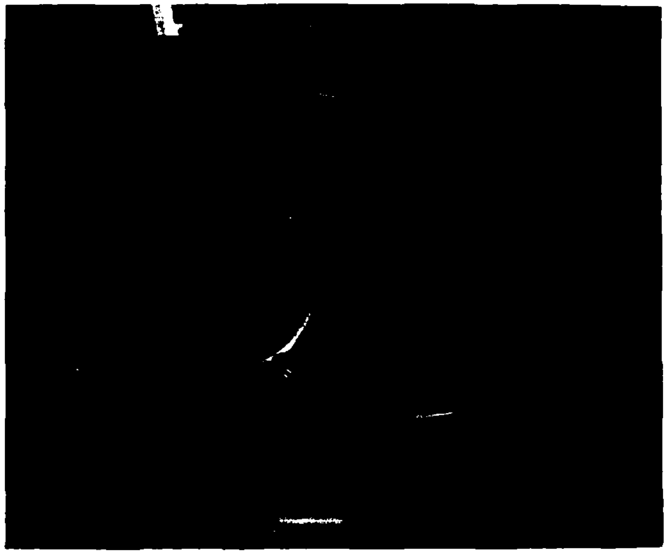
0-79-17 RESIDUE AT $275^{o}C$/30 HOURS

0-79-17 RESIDUE AT $300^{o}C$/15 HOURS

Figure 17. Scanning Electron Microscope (SEM) Photographs of 0-79-17 Residue Obtained from the MCRT at 275°C for 30 Hours and 300°C for 15 Hours

0-79-17 RESIDUE AT 275°C/30 HOURS TEST #11





0-79-17 RESIDUE AT 275°C/30 HOURS TEST #8

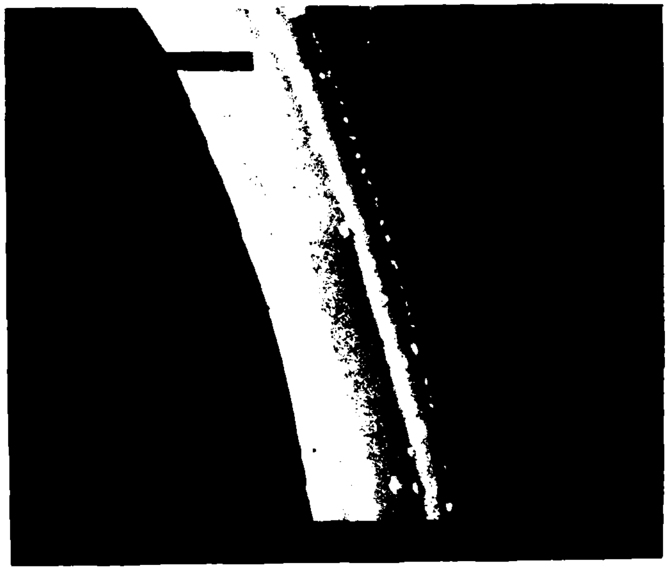
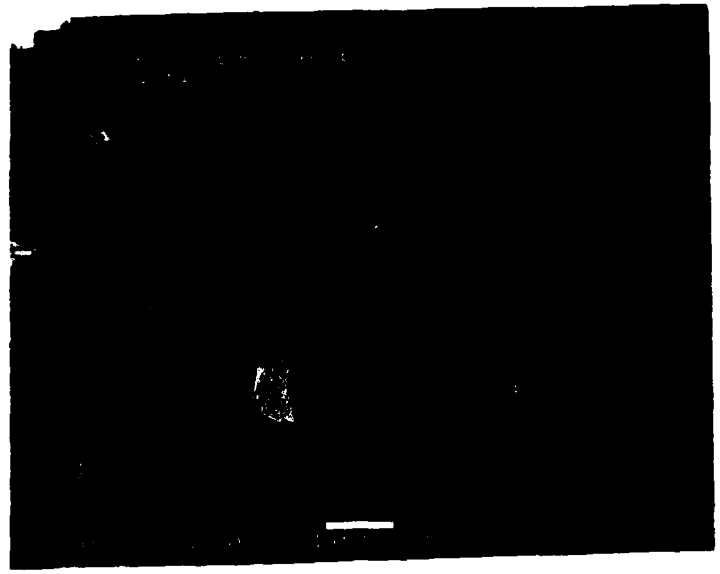


Figure 18. Scannin g Electron Microscope (SEM) Photographs of 0-79-17 Residue Obtained from the MCRT-100 at 275°C for 30 Hours and Different Tests (#8 and #11) at 100 and 1000 Magnification

TEMPERATURE: ca 385°C

TEMPERATURE: ca 315°C

Figure 19. Carbon Residue from J57-P29 100-Hours Engine Test - Lubricant No. 0-82-14



TEMPERATURE: ca 325°C

TEMPERATURE; ca 191°C

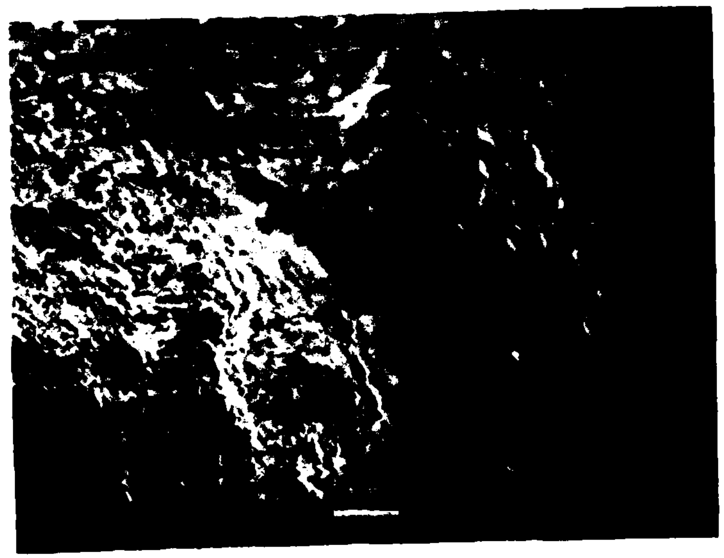


Figure 20. Carbon Residue from J57-P29 100-Hours Engine Test - Lubricant No. 0-82-14

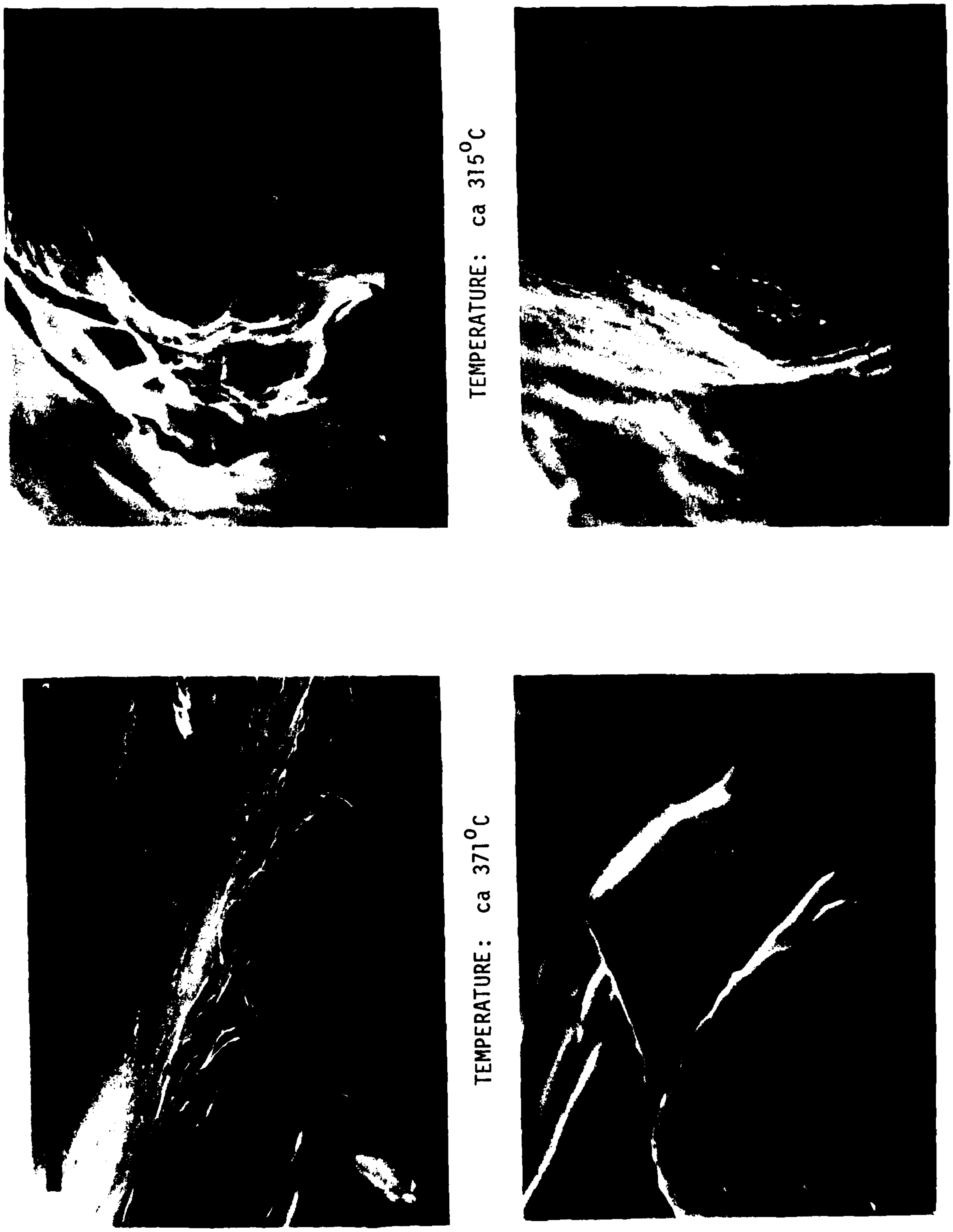


Figure 21. Carbon Residue from J57-P29 100-Hours Engine Test - Lubricant No. 0-82-14

a. MCRT-100 Timer

If during a test the gas supply or the power goes off, the controller goes into "remote hold" or shows "flag" (the test stops) so that the operator is aware that there was a gas or power failure; but the controller does not stop, count or show the test time. The power went off very often during long-term tests, which ran over nights and weekends and the test results could not be used because the time when the power breakdown was not known, and so the exact test time could not be calculated. To avoid this situation, Alcor Inc., San Antonio, TX, developed a prototype unit which was hooked up between the gas supply, MCRT and timer. The timer and the MCRT were plugged into the unit. On the unit there is a switch which has two positions: run (forward) and reset (backward). In run position, the system will run until either a power failure occurs or the gas pressure drops below about 3 psi. When either event occurs, the timer will stop. If either the power or the gas is then restored, nothing will start again until the switch is moved to reset. By setting the timer to zero at the beginning of the test run, many test data for temperature profiles could be saved, because the exact time until the failure occurred was known.

7. COMPARISON OF TEST RESULTS CONDUCTED UNDER DIFFERENT CONDITIONS AT 275°C FOR 30 HOURS AND 300°C FOR 15 HOURS

Tests under different conditions were conducted to determine how strongly changes in test variables affect results in the amount of residue, repeatability, morphology, and C-H-N content.

a. Data from tests with two different ramp time/temperatures at 275°C for 30 hours and 300°C for 15 hours

The different test programs are shown in Figure 3 and Table 6a.

TABLE 10

TESTS WITH TWO DIFFERENT RAMP TIME/TEMPERATURES AT
275°C FOR 30 HOURS AND 300°C FOR 15 HOURS

	RESIDUE (WT%) *			
	OIL CODES			
TEST TIME/ TEMPERATURE	0-82-14	0-72-11	0-79-17	0-75-1
30 hrs/275°C**	18.39 ± 0.58	13.56 ± 0.43	9.84 ± 0.14	9.02 ± 0.60
30 hrs/275°C***	18.19 ± 0.17	12.93 ± 0.37	9.85 ± 0.15	9.46 ± 0.27
15 hrs/300°C**	13.83 ± 0.41	13.19 ± 0.34	12.88 ± 0.27	10.65 ± 0.18
15 hrs/300°C***	13.04 ± 0.40	12.53 ± 0.70	12.56 ± 0.47	10.63 ± 0.73

* All data of Table 10 (Residue in %) are mean of three tests

** Test Program: (according to Table 6a) 1/2 hr to 150°C, 1 hr at 150°C, 1/2 hr to 275°C or 300°C, 30 hrs at 275°C or 300°C for 15 hrs.

*** Test Program: (according to Figure 3) 1/2 hr to 275°C or 300°C (high purge on), 30 hrs at 275°C or 15 hrs at 300°C.

A comparison of the data from Table 10 shows that the initial ramp process has no effect on the amount of residue obtained. The repeatability was excellent with both test conditions. SEM pictures and C-H-N analyses of residue from lubricant 0-82-14 were obtained to see if a difference in the morphology and the C-H-N content between the obtained residue from the different ramp times/temperatures could be observed. Figures 22 (Test #38) and 23 (Test #34) present SEM pictures of residue from lubricant 0-82-14 at the different test conditions. As it can be seen, there is no difference in the morphology. The C-H-N analyses (Table 21) also show no difference in the C-H-N content at both conditions. Therefore, since no difference in the amount of residue, the repeatability, the morphology, and the C-H-N content could be observed, subsequent tests were conducted with a 1/2 hour heating period to 275°C or 300°C.

O-82-14 ARE COLLECTED AFTER A 100-HOUR J57-P29 ENGINE TEST AND TESTED AT 275°C & 30 HOURS TEST TIME IN MCRT-100.

O-82-14 FRESH OIL TESTED AT 275°C & 30 HOURS TEST TIME IN THE MCRT-100.

Figure 22. Scanning Electron Microscope (SEM) Photographs of Carbon Residue Obtained from MCRT-100 at 275°C for 30 Hours Test Time - Lubricant O-82-14

30 HOURS AT TEMPERATURE 275^{o}C

Figure 23. Scanning Electron Microscope (SEM) Photographs of Carbon Residue Obtained from MCRT-100 at 275°C for 30 Hours Test Time - Lubricant 0-82-14

b. Measurements of the Air and Nitrogen Flow Rates Through the MCRT Oven and Comparison of Residue Data Indicated at Different Airflow Rates (150, 600 and 1000 cc/min)

(1) Measurements of the air and nitrogen flow rate through the MCRT oven.

The different flow rates through the oven were measured by connecting the MCRT chimney to a wet test meter. The oven temperature was 275°C and the pressure on the MCRT gauge was exactly 20 psig.

TABLE 11

ACTUAL FLOW RATE MEASUREMENTS CONDUCTED WITH A WET TEST METER

FLOW RATE ADJUSTED WITH THE MCRT MICROPROCESSOR	ACTUAL FLOW RATE MEASURED WITH THE WET TEST METER
150 cc/min air at 20 psig	144 cc/min
600 cc/min air at 20 psig	605 cc/min
150 cc/min nitrogen at 20 psig	142 cc/min
600 cc/min nitrogen at 14 psig	430 cc/min
1000 cc/min air at 34 psig*	1000 cc/min

* Some tests were conducted at 1000 cc/min airflow, and therefore, the microprocessor was programmed for high purge (600 cc/min) and then the pressure on the MCRT gage was increased until 1000 cc/min was measured with the wet test meter.

The data indicate that there is only an insignificant difference between the adjusted and the actual flow rate through the system at 600 cc/min nitrogen flow. The flow rate through the MCRT system is fixed by internal needle valves which normally deliver 150 cc/min or 600 cc/min at 20 psig on the MCRT gauge.

During the measurements with 600 cc/min nitrogen flow, the pressure on the MCRT gauge dropped from 20 psig to 14 psig and after which the actual flow rate was measured to be 430 cc/min. After increasing the pressure on the gauge from 14 psig to 20 psig during the test, the actual flow rate was measured to be 610 cc/min. The only explanation for the drop in gauge pressure was that the nitrogen regulator didn't hold the pressure constant. After replacing the nitrogen regulator with a new one, only a slight pressure drop was measured. Thereafter, pressure dropped from 20 psig to 19.5, when using nitrogen or air.

TABLE 12

COMPARISON OF DATA FROM TESTS WITH DIFFERENT AIRFLOW RATES (150 AND 600 CC/MIN AND 1000 CC/MIN)

	RESIDUE WT%			
	OIL CODES			
AIRFLOW RATE (CC/MIN)	0-82-14	0-72-11	0-79-17	0-75-1
*150 cc/min at 20 psig	18.19 ± 0.17	12.93 ± 0.37	9.85 ± 0.15	9.46 ± 0.27
*600 cc/min at 20 psig	18.72 ± 0.30	13.92 ± 0.52	11.65 ± 1.69	9.77 ± 0.31
*1000 cc/min at 34 psig	17.91 ± 0.64	13.02 ± 0.80	8.92 ± 0.99	10.31 ± 0.61
**150 cc/min at 20 psig	13.40 ± 0.57	12.54 ± 0.70	12.56 ± 0.47	10.63 ± 0.73
**600 cc/min at 20 psig	13.75 ± 0.56	12.76 ± 0.67	13.34 ± 0.61	10.99 ± 0.45
**1000 cc/min at 34 psig	13.41 ± 0.23	13.37 ± 0.36	13.37 ± 0.30	11.37 ± 0.25

* Test Program: 1/2 hr to 275°C and 30 hrs at 275°C (See Figure 3).
** " " 1/2 hr to 300°C " 15 hrs at 300°C " " ".

The data indicate that increasing airflow rates (150 cc/min versus 600 cc/min) results in an increased amount of residue (2.5 to 16% at 275°C for 30 hours and 3.7 to 8.7% at 300°C for 15 hours). However, the amount of residue decreased slightly at an airflow rate of 150 cc/min versus 1000 cc/min. Lubricant 0-75-1 B/S is the only lubricant which increased the amount of residue with increased airflow rates (600 cc/min and 1000 cc/min). Lubricant 0-75-1 B/S is the basestock from 0-79-17 and has no additive package, which may be the reason for this performance. It also can be seen that the repeatability at higher airflow rates versus a lower rate of 150 cc/min is not as good. Therefore, all the following tests were carried out at 150 cc/min.

c. Comparison of Test Data from Tests With Fewer Than 12 Vials in the MCRT Oven

TABLE 13

DATA FROM TESTS WITH 3, 5, AND 12 VIALS IN THE MCRT OVEN WITH LUBRICANT 0-72-11 AT 275°C FOR 30 HOURS

POSITION OF THE VIALS	RESIDUE WT%*
1 to 12	13.59 ± 0.60
2, 4, 6, 8 and 12	13.53 ± 0.23
4, 6 and 8	14.16 ± 0.23
1, 3 and 5	12.50 ± 0.24

* Means of two tests.

Repeatability tests with 3, 5, and 12 vials in the MCRT oven with a MIL-L-7808 lubricant at 275°C for 30 hours indicate that there is only a slight difference in the amount of residue when using 5 (in positions 2, 4, 6, 8 and 10) or 12 vials. With only 3 vials mounted in the oven at a time, the amount of residue was higher in position 4, 6 and 8 and lower in position 1, 3 and 5. These data indicate that the amount of residue generated was dependent upon the position of the vial. Therefore, it is recommended that at least 5 vials (positions 2, 4, 6, 8, and 10) or more should always be used in the MCRT-100 oven.

d. High Temperature Cycle to Prevent Plugging of the MCRT Oven.

During the investigation, the pipe connecting the MCRT-100 oven with the condensate trap and the chimney was totally plugged with solid residue after several determinations.

To prevent plugging, the following high temperature cycle should be executed after every 6th test. Heat the MCRT oven in 1/2 hour to 580°C (no oil samples in the oven) and hold this temperature for six hours. Then allow the oven to cool to room temperature. High purge (600 cc/min airflow) should be on during the entire test. This will not only prevent the oven from plugging up but also will prevent residue from accumulating on the surface of the oven lid. The oven lid is a special gravity seal lid, which has a spherical plug that seals the oven and is insulated to maintain a high surface temperature so that the vapor of the lubricants tends not to collect on that surface. But even the special lid did not prevent a layer of residue from forming after some tests. The residue on the oven lid must be removed before each test, or it may be possible that some residue would fall into an oil sample and give significantly higher results.

e. Tests with 1/2 Dram Vials of Different Length (3.6 Cm and 7.2 Cm)

TABLE 14

COMPARISON OF DATA FROM TESTS WITH 1/2 DRAM VIALS OF DIFFERENT LENGTH (3.2 CM VERSUS 7.2 CM) AT 300°C FOR 15 HOURS

RESIDUE WT%

	LUBRICANT			
LENGTH OF THE VIALS	0-82-14	0-72-11	0-79-17	0-75-1
7.2 cm	20.65 ± 1.39	16.95 ± 0.93	16.82 ± 0.97	14.79 ± 1.08
7.2 cm	20.18 ± 1.36	17.07 ± 1.17	16.85 ± 1.22	14.95 ± 1.09
MEAN AND STANDARD DEVIATION OF TWO TESTS	20.42 ± 0.33	17.01 ± 0.08	16.84 ± 0.02	14.87 ± 0.11
3.2 cm	13.01 ± 0.73	12.33 ± 0.30	12.31 ± 0.28	10.41 ± 0.40
3.2 cm	13.13 ± 0.53	11.97 ± 0.28	12.27 ± 0.24	10.09 ± 0.31
MEAN AND STANDARD DEVIATION OF TWO TESTS	13.07 ± 0.08	12.15 ± 0.25	12.29 ± 0.02	10.25 ± 0.23

The data show that the amount of residue increased between 31% to 56% when using 1/2 dram vials which had a length of 7.2 cm instead of the standard 3.2 cm. The data also indicate that differentiation between lubricants meeting the same specification is much better using the longer 1/2 dram vials. One candidate MIL-L-7808 nearly plugged the longer vials completely and showed the most amounts of residue with 20.65 ± 0.33%. The only disadvantage using the long vials was that the standard deviation as compared to the regular 1/2 dram vials (3.6 cm) was not as good, but before a repeatability statement can be made, additional data will be required. It was decided to conduct more tests with the long 1/2 dram vials but that was not possible because the vials were not available during this work. It is suggested that more tests be carried out with different lubricants, and the longer 1/2 dram vials to see if they should replace the standard vials.

f. MCRT-100 Residue Obtained from Stressed Lubricants According to FTM 5307.1

TABLE 15

MCRT-100 RESIDUE OBTAINED FROM FRESH AND STRESSED LUBRICANT 0-79-17 AT 300°C FOR 15 HOURS. LUBRICANT 0-79-17 WAS STRESSED ACCORDING TO FTM 5307.1 TEST METHOD (CORROSION AND OXIDATION STABILITY OF SYNTHETIC GAS TURBINE LUBRICANTS) FOR 96 HOURS AT 200°C

	RESIDUE WT%
Fresh Oil	12.56 ± 0.47
Stressed Oil (Batch No 84)	13.83 ± 0.46

The data indicate that stressed 0-79-17 produces 10.1% more residue than the fresh lubricant.

g. Data from Formulation Mixtures of Fresh Lubricants Meeting MIL-L-7808J specification.

TABLE 16

MIXTURES OF LUBRICANTS MEETING MIL-L-7808J SPECIFICATION TESTED AT 300° FOR 15 HOURS IN THE MCRT

MIXTURE RATIOS in WT%	RESIDUE WT%
100 (0-82-14)	13.72 ± 0.68
80/20 (0-82-14/0-79-17)	13.89 ± 0.35
50/50 (0-82-14/0-79-17)	13.78 ± 0.44
20/80 (" ")	13.30 ± 0.32
100 0-79-17	12.56 ± 0.47
100 (0-72-11)	12.78 ± 0.60
80/20 (0-72-11/0-82-3)	12.60 ± 0.41
50/50 " "	10.67 ± 0.36
20/80 " "	9.32 ± 0.40
100 0-82-3	8.49 ± 0.18

The data from Table 16 indicate that the amount of residue is not significantly higher for the mixtures investigated. It is suggested that more tests be carried out with lubricant mixtures.

h. Data on residue obtained from the MCRT-100 at 275°C for 30 hours and 300°C for 15 hours was compared with the data from the static coker tester, the Bearing Deposition Tester (BRG DEP), and the Wright Air Development Center Deposition Tester (WADC DEP).

The test results from Table 17 are presented in Figures 15 and 16. It can be seen that lubricants TEL-5027, TEL-5028, and TEL-5029 show the highest amount of coke deposits in the MCRT-100 for all 3 and 5 cSt lubricants tested. The explanations for this could be due to the lower evaporation loss of the 5 cSt lubricants compared to the 3 cSt lubricants, and due to the higher amount of additives used in the 5 cSt lubricants meeting D.ENG.RD 2497 specification compared to the 5 cSt lubricants meeting MIL-L-23699C specification. No test data were available from BRG and WADC deposition tests for these lubricants.

Lubricants 0-71-6 and 0-77-15 are 5 cSt oils meeting MIL-L-23699C specification. These lubricants had the least amount of residue of the 5 cSt lubricants tested, but showed approximately 5% more residue compared to the highest amount of residue with 3 cSt lubricants. The bearing deposition #20 for 0-77-15, however, indicates that lubricant 0-77-15 had the lowest average amount of residue for all 3 and 5 cSt lubricants in bearing deposition tests. 0-71-6 gives with a deposition #37 a medium range in coke deposits in bearing deposition tests.

Lubricant 0-82-14 (3 cSt) had the highest average coke deposit (18.39 ± 0.58) in the MCRT-100 for all 3 cSt lubricants and had a medium range in coke deposits in the bearing deposition tests (deposition #38). In WADC deposition tests, this lubricant had the second lowest amount of coke deposits with a deposition number of 0.2. Lubricant 0-82-14, however, is the only 3 cSt lubricant evaluated which did not pass the J57 full-scale engine test. It passed all test requirements according to MIL-L-7808J and even passed the J57 engine simulator 100-hour test, but did not pass the J57 engine test because of excessive deposits. The previous data indicate that the MCRT-100 was the only deposition tester which identified lubricant 0-82-14 as a high coking oil.

Lubricant 0-82-2 (3 cSt meeting MIL-L-7808H) showed the second highest amount of residue in the MCRT-100 from the 3 cSt lubricants. The deposition #35 gave a medium range in coke deposits with the BRG DEP and showed the highest amount of deposits (deposition #0.8) with the WADC DEP.

Lubricant 0-72-11 (0-79-16 but a different batch) is a 3 cSt lubricant meeting MIL-L-7808J. Both lubricants give a high medium range in coke deposits in the MCRT-100 for the 3 cSt lubricants, and the deposition #49 indicates the highest amount of coke deposits in the BRG DEP and the second highest amount of coke deposits (deposition #0.7) in the WADC DEP from all 3 and 5 cSt lubricants.

Lubricant 0-78-9 (3 cSt meeting MIL-L-7808J) showed a medium range in coke deposits in the MCRT-100 and had a medium range in coke deposits in the BRG DEP with a deposition #36. It gave the third highest amount of deposits (deposition #0.6) in WADC deposition tests.

Lubricant 0-79-17 (3 cSt meeting MIL-L-7808J) gives a low range in coke deposits at 275°C for 30 hours and a medium range in coke deposits at 300°C for 15 hours. The deposition #30 indicates the second lowest amount of residue in BRG DEP tests and that lubricant gives the lowest amount of deposits in WADC deposition tests (deposition #0.1).

Lubricant 0-75-1 (basestock from 0-79-17) gives a low range in coke deposits in the MCRT. Between basestock (0-75-1) and full formulation (0-79-19), there is no significant difference in the amount of residue.

Lubricant 0-78-8 (3 cSt meeting MIL-L-7808J) had the second lowest amount of residue in the MCRT at 275°C for 30 hours, but the highest amount of residue at 300°C for 15 hours. It had the lowest amount of deposits (deposition #27) for the 3 cSt oils in the BRG DEP and also gives the lowest deposition # (0.1) in the WADC DEP.

TABLE 17

LUBRICANT COKE-FORMING TENDENCIES WITH THE MICRO CARBON RESIDUE TESTER (MCRT-100), THE BEARING DEPOSITION TESTER (BRG DEP) [5], THE WRIGHT AIR DEVELOPMENT CENTER DEPOSITION TESTER (WADC DEP) [4], AND THE STATIC COKER DEPOSITION TESTER [3]

OIL CODE	RES WT% (+) MCRT-100	RES WT% (+) MCRT-100	STATIC COKER DEPOSITION TSTR	BRG DEP (MIL-L-7808J)			WADC DEP (MIL-L-7808J		
5 cSt Lubricants	@ 275°C for 30 Hrs	@ 300°C for 15 Hrs	Res in mg (310°C)	DEP#	%VISC 100°F	TAN	DEP#	%VISC 100°F	TAN
TEL-5028	27.78 ± 0.36	27.47 ± 0.23	41.2	--	--	-	--	--	-
TEL-5029	27.31 ± 0.32	26.39 1 0.40		--	--	-	--	--	-
TEL-5027	26.77 ± 0.19	25.07 ± 0.30		--	--	-	--	--	-
0-77-15	24.17 ± 0.35	24.17 ± 0.75		20	--	-	--	--	-
0-71-6	21.15 ± 0.32	21.15 ± 0.32	29.5	37	--	-	--	--	-
3 cSt Lubricants									
0-82-14*	18.19 ± 0.17	13.82 ± 0.68		38	13	1.0	0.2	60	9.3
0-82-2	17.70 ± 0.20	13.01 ± 0.68		35	7	0.2	0.8	31	6.0
0-72-11**	12.93 ± 0.37	12.78 ± 0.60		49	7	0.2	0.7	57	9.5
0-79-16	12.41 ± 0.22	12.59 ± 0.16		49	7	0.2	0.7	57	9.5
0-78-9	10.36 ± 0.57	12.27 ± 0.18		36	15	0.4	0.6	21	1.6
0-79-17	9.88 ± 0.24	12.56 ± 0.47	13.8	30	10	0.8	0.1	16	1.8
0-75-1 B/S***	9.46 ± 0.27	10.94 ± 0.68		--	--	--	--	--	--
0-78-8	8.80 ± 0.53	13.76 ± 0.22		27	13	0.8	0.1	18	0.9
0-82-3	8.09 ± 0.24	8.49 ± 0.18		40	14	0.5	0.4	56	6.2

* 0-82-14 Average of five tests.
** 0-72-11 " " " " .
*** 0-75-11 B/S Base Oil for 0-79-17.
(+) The test data are means of three tests.

Lubricant 0-82-3 (3 cSt meeting MIL-L-7808J) shows the lowest amount of residue in the MCRT-100. The deposition #40 indicates the second highest amount of residue in the BRG DEP, and the deposition #0.4 gives a medium range of deposits in the WADC DEP.

The data from Table 17 indicate that the test results (amount of residue in wt% or deposition numbers) from the MCRT, the BRG DEP (Reference 5), and the WADC DEP (Reference 4) are not comparable. For example, lubricant 0-82-14 shows the highest amount of residue in the MCRT, a medium amount of residue in the BRG DEP, and a low amount of residue in the WADC DEP. The most probable explanation is the difference in the test conditions. The MCRT-100, however, is the only test device which pointed out that lubricant 0-82-14 is a high coking 3 cSt oil which was later verified in a full-scale engine test which failed due to excessive carbon deposition. Table 17 shows also that lubricant 0-82-3 had the lowest amount of residue at 275°C for 30 hours and had the highest amount of all 3 cSt oils at 300°C for 15 hours. The explanation for this could be that of lubricant formulation. Certain constituents of some lubricants may evaporate before those of another lubricant, thereby causing lower remaining deposits.

The static coker deposition tester and the MCRT-100 differentiate lubricants meeting MIL-L-7808J, MIL-L-23699C, and D.ENG.RD 2497 in a similar manner. This is apparently due to the similarity of test conditions. In both tests a major effect is volatilization. One difference is that in this static coker, lubricant/metal contact occurs throughout the test. It would be beneficial to determine if ranking lubricants from a specification is the same using the MCRT and the Static Coker test. Therefore, it is suggested that comparative tests with the MCRT and the Static Coker (Reference 3) be done.

8. SEM-PICTURES AND CARBON-HYDROGEN-NITROGEN (C-H-N) ANALYSES OF RESIDUE COLLECTED FROM THE MCRT-100, THE J57 ENGINE AND THE J57 ENGINE SIMULATOR

This is to see how the residue from the MCRT-100 at different times and temperatures can be compared with residue from the J57-P29 engine and the J57 engine simulator. Some residue was collected from different engine parts after a 100-hour J57-P29 engine test with lubricant 0-82-14 and after a 100-hour J57 engine simulator test with lubricant 0-79-17. The above residue and MCRT-100

residue were then compared under the SEM and C-H-N. The analysis obtained is discussed in the following:

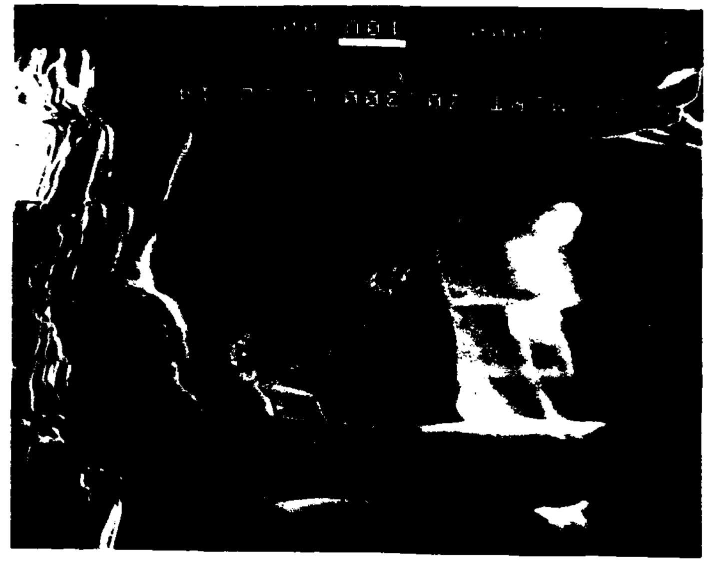
Figures 19, 20, and 21 present SEM-pictures of lubricant 0-82-14 residue collected from the following J57 engine parts.

1.	Tower shaft	Temp. 315 - 330°C
2.	Diffuser case	" 191°C
3.	#5 Bearing seal support	" 315°C
4.	#6 Bearing breather	" 380°C
5.	#6 Bearing sump cover	" 371°C
6.	#6 Bearing sump	" 315°C

It can be seen (Figures 17 to 25) that there is a significant difference between residue from the J57 engine simulator, the J57 engine, and the MCRT-100. The residue from the MCRT is very smooth in most cases and is similar to the appearance of a layer of paint when examined by SEM. It is very hard and breaks very easily. Only test #37 (Figure 21) shows a difference in the morphology at 1000 X magnification. This oil was collected after a 100-hour J57 engine test and was then tested in the MCRT-100. It may contain wear debris and this may be the reason for the difference in the morphology. The formation of solid residue in the J57-P29 engine simulator or the J57 engine occurs in widely differing, irregular forms, from thin layers (soft in consistency) to very thick layers (hard crusts). Some of the residue looks as if it were formed in the vapor phase (#6 breather). The SEM-pictures of carbonaceous residue generated from the J57 engine simulator, the J57 engine, and the MCRT-100 illustrate the differences observed and indicate that direct comparisons are difficult. The comparison of the test conditions in a turbine engine and the MCRT-100 shows that the same residue should not obviously be expected. In a turbine engine, one has static and dynamic processes and many parameters which have an influence on the formation and the amount of residue, but in the MCRT one has only static conditions. It is believed that the following parameters influence the formation of deposits in turbine engines.

1. Oil temperature (bulk oil temperature, temperature of the oil which is in contact with the hot metal surface and the temperature of the oil wetted metal surface).

30 HOURS AT TEMPERATURE 300°C



30 HOURS AT TEMPERATURE 330°C

Figure 24. Scanning Electron Microscope (SEM) Photographs of Carbon Residue Obtained from MCRT-100 at 300 and 330°C for 30 Hours

#5 BEARING HOUSING: TEMP ca 340°C

#5 BEARING CARBON SEAL RING ASSEMBLY: TEMP 330-340°C

Figure 25. SEM Pictures of Carbon Residue Obtained from J57 Engine Simulator 100-Hours Engine Te-t, Lubricant No. 0-79-17

2. The oil residence time (volume to surface ratio)
3. The lubricant/oxygen ratio.
4. Catalytic reactions of metals (metal/lubricant).
5. The lubricant state (vapor, droplets, film).
6. Moisture.
7. Filtration.
8. Formulation (viscosity, acid number, vapor pressure, and different additive packages).

The major effect during a test with the MCRT is apparently volatilization of the lubricant. Substantial oxidation and thermal degradation also occurs, which leads to the formation of residue in the glass vials. But the degradation of the lubricant in the MCRT is not very realistic because the evaporated oil is caught in the condensate trap and does not return to the remaining oil in the sample vials. It is also known that the condensate, which returns to the bulk oil in an engine, has an effect on viscosity, acidity and the degradation of the lubricant. The residue remaining after complete evaporation in the MCRT oven consists mainly of deposits from the breakdown of the oil and includes nonvolatile additive residual materials. All the above observed differences indicate that it is very difficult and may be impossible to simulate test conditions of a gas turbine engine in a single laboratory test.

Figure 14 presents residue data from the MCRT-100 as a function of test temperature at constant test time (30 hours) with four oils (0-82-14, 0-72-11, 0-79-17, and 0-75-1 B/S). The data indicate that oil 0-82-14 has more solid residue after tests at different temperatures than 0-72-11, 0-79-17 and 0-75-1 B/S. Even the formation of residue in the vials was different. Oil number 0-82-14 formed an amount of residue on the upper edge of the vials, which nearly plugged up the 1/2 dram vials. Only oil number 0-72-11 showed a similar tendency, but the amount of residue was not as high. Lubricant 0-82-14, however, is the only oil which did not pass the J57 full scale engine test. It passed all the tests required according to the MIL-L-7808J specification and even passed the J57 engine simulator 100-hour test, but did not pass the J57 engine test because of excessive deposits. Oil number 0-75-1 B/S gives the lowest remaining residue and the highest volatility. 0-75-1 B/S is the base oil from 0-79-17 and has no additive package which could improve the thermal oxidative stability or could remain after the breakdown of the oil. But the data also indicate only slight difference in the amount of residue between the base oil 0-75-1 B/S and the full formulated 0-79-17.

Table 18 compares the deposit ratings of three lubricants tested in the J57 engine, the J57 engine simulator, and the MCRT-100.

TABLE 18

COMPARISON OF DEPOSIT RATINGS OF THREE LUBRICANTS TESTED IN THE J57, THE J57 ENGINE SIMULATOR AND THE MCRT-100

LUBRICANT NO.	DEPOSIT RATING J57 ENGINE SIMULATOR	MCRT-100
0-82-14**	19.60	18.39
0-72-11	17.60	12.92
0-79-17***	14.40	9.97
0-75-1 B/S*	--	9.02

* 0-75-1 B/S is the base oil from 0-79-17.
** 0-82-14 did not pass the engine test because of excessive deposits.
*** 0-79-17 was cleaner than 0-72-11 after a 100-hour J57 engine test.

Tables 19, 20 and 21 give C-H-N data of 0-82-14 carbon residue collected after a J57 100-hour engine test, 0-79-17 carbon residue collected after a J57 100-hour engine simulator test, and residue from lubricants 0-79-17; 0-82-14 obtained in the MCRT-100. Table 20 presents 0-82-14 residue collected from the J57 engine and indicates a slight difference in the wt% of C-N-H content between residue from different engine parts. The carbon content ranges from 70.25 wt% to 73.63 wt%, the H content from 4.96 to 6.87 wt% and the N content from 1.05 to 2.28 wt%. The unanalyzed residue ranges from 19.16 to 22.19 wt% and may contain metals, oxygen, phosphorus, and sulfur. Only the residue of #6 bearing breather should not contain free metals as the rest of the residue from the J57 engine because this residue should be formed in the vapor phase. From Table 21 (0-82-14 carbon residue obtained in the MCRT) only #36 (30 hours at 330°C, which actually is 344°C) shows results similar to #6 bearing breather. All other data from Table 21 show quite a difference compared to the data from the J57 engine (Table 20). Table 21 also indicates that there is no difference in the C-H-N content (Tests #34 and #38) if a different ramp time/temperature in the MCRT was used. The C-H content of residue (Lubricant 0-82-14) from the MCRT at 300°C for 30 hours (Table 21, #33) compared to residue at 275°C for 30 hours test time (Table 21, #34) is very low. Why the C-H content went up at 330°C for 30 hours test time (Table 21, #36) cannot be explained and needs further investigation. The residue (Lubricant 0-79-17) from the J57 engine simulator (Table 19) shows very low H

contents (3.26 - 3.47) and a very high N content (4.42) compared to a H-N content of 6.14 and .93 for the MCRT-100 residue. The above C-H-N analysis indicate a significant difference in the C-H-N content of residue from the J57 engine, the J57 engine simulator, and the MCRT-100 and confirm that direct comparisons are difficult.

TABLE 19

C-H-N ANALYSIS OF 0-79-17 CARBONACEOUS RESIDUE FROM THE MCRT-100 AT DIFFERENT TIMES/TEMPERATURES AND OF 0-79-17 RESIDUE COLLECTED AFTER A 100-HOUR J57 ENGINE SIMULATOR TEST

WT%

SAMPLE	TEST TIME HRS	TEST TEMP. °C	C-MEAN		H-MEAN		N-MEAN		UNANALYZED**
#24*	30	275	65.59	65.84	5.61	5.73	0.86	0.93	27.5
			66.09		5.84		1.00		
#28*	96	275	69.17	69.21	6.08	6.12	0.96	0.95	23.72
			69.26		6.17			0.94	
#56*	10	300	70.57	70.45	6.11	6.14	1.45	1.16	22.25
			70.33		6.17		0.87		
#57*	15	300	69.89	70.10	5.51	5.60	1.23	1.13	23.16
			70.32		5.68		1.04		
#5 Brg Carbon Seal	100	330-340	67.63	66.78	3.28	3.26	4.42	4.42	24.54
			67.92		3.24			4.42	
#5 Bearing Housing	100	330-340	68.72	67.89	3.47	3.47	4.40	4.4	24.24
			67.50		3.44		4.43		
			67.45		3.40		4.37		

** The unanalyzed residue from the J57 engine simulator probably consist of metals, oxygen, phosphorus, and sulfur. The unanalyzed residue from the MCRT-100 probably consist of oxygen, sulfur, and phosphorus.

* #24, 28, 26 and 57 are same test procedure but different times and temperatures.

Test Procedure: 1/2 hour heating period to 150°C, hold 1 hour, 1/2 hour to X-°C and then X-hours test times.

TABLE 20

C-H-N ANALYSIS OF O-82-14 CARBONACEOUS RESIDUE COLLECTED FROM J57 100-HOUR TEST ENGINE

WT%

SAMPLE	C	MEAN	H	MEAN	N	MEAN	UNANALYZED*
Tower Shaft	73.26	73.61	4.82	4.96	2.29	2.28	
	74.02		5.09		2.26		19.16
Diffuser Case	72.20	71.93	4.40	4.95	2.37	2.22	
	71.65		5.50		2.06		20.91
#5 Brg	69.88	70.25	5.48	5.50	2.04	2.12	
Seal Support	70.61		5.51	2.20			22.14
#6 Brg	71.53	71.45	5.33	5.32	1.12	1.05	
Breather	71.36		5.31		0.98		22.19
#6 Brg	71.14	71.36	6.20	6.30	1.14	1.14	
Sump	71.58		6.40		1.14		21.20
#6 Brg Cover	78.89	75.35	7.18	6.87	1.78	1.73	16.06
	77.81		6.56		1.67		

*Unanalyzed residue probably consist of metals, oxygen, phosphorus, and surfur.

TABLE 21

C-H-N ANALYSIS OF O-82-14 CARBONACEOUS RESIDUE FROM MCRT-100

WT%

SAMPLE	TEST TIME HRS	TEST TEMP. °C	C-MEAN		H-MEAN		N-MEAN		UNANALYZED*
#34	30	275	68.88	68.71	5.37	5.38	0.83	0.80	25.11
			68.54		5.38		0.76		
#37	"	"	69.25	69.25	3.99		0.80		25.96
#38	"	"	68.50	68.45	5.41	5.31	0.48	0.49	25.74
			68.40		5.24		0.49		
#33	"	300	57.96		2.05		0.91		39.08
#36	"	330	71.85	71.28	4.68	4.54	0.88	0.81	23.38
			70.71		4.40		0.73		

#38 — 1/2 hour heating period to 275°C and then 30 hours test time.
#33 to 37 — 1/2 hour heating period to 150°C, hold for 1 hour, then in 1/2 hour to 275, 300 and 330°C and after that 30 hours test time.
#37 — Aged lubricant from the 100-hour J57 engine test.

*Unanalyzed residue from the MCRT-100 probably consists of oxygen, sulfur, and phosphorus.

9. PROPOSED TEST PROGRAM FOR TESTING 3 AND 5 cSt SYNTHETIC GAS TURBINE LUBRICANTS

The following MCRT program was chosen and is proposed for testing 3 and 5 cSt lubricants considered for MIL-L-7808J, MIL-L-23699C, and D.ENG.RD 2497 specification use. The test procedure is given in Section II.

TABLE 22

PROPOSED TEST PROGRAM FOR 3 AND 5 cSt LUBRICANTS (INITIAL SETPOINT, SEGMENT ENDPOINT, TIME OF SEGMENT, CONTROL TUNING CONSTANTS, AND EVENTS)

PROGRAM AND SEGMENT	SEGMENT END POINT °C	TIME OF SEGMENT IN HRS.	EVENTS* 1	2	3	4
1-1	0	0	ON	OFF	OFF	OFF
1-2	300	0.5	OFF	ON	ON	ON
1-3	300	30	OFF	OFF	OFF	ON
1-4	0	0	OFF	OFF	OFF	ON
1-5	0	0	OFF	OFF	OFF	ON
1-6	0	0	OFF	OFF	OFF	ON
1-7	0	0	OFF	OFF	OFF	ON
1-8	0	0	OFF	OFF	OFF	ON
3-1	0	0	OFF	OFF	OFF	OFF

Control tuning constants are:
Initial set point = 0
Cycle time = 2
Out 1 limit = 50
Proportional band = 10
Reset = 1
Rate = 0.1

* Events are:

1 = Remote reset
2 = Reset inhibit
3 = High purge on
4 = Program on

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

1. CONCLUSIONS

The foregoing evaluation showed that the MCRT-100 has potential use as a cost effective laboratory test to differentiate lubricants regarding tendency to form coke deposits under certain test conditions prior to engine test. The investigation also showed that the repeatability with the test unit is excellent. The parameters that influence the formation of deposits (airflow through the system, test time and temperature) could be adjusted very easily with the microprocessor and were constant during tests.

The actual lubricant sample temperature versus the display temperature on the MCRT-100 was 10-14°C higher. The test temperature in the oven during tests was very consistent.

Comparisons of collected residue from the MCRT-100, the J57 engine simulator, and the J57 engine showed differences in the residue morphology and in C-H-N content. These differences can be explained as due to the different conditions encountered by a lubricant in the different tests. In an engine, one has static, dynamic and many other parameters that influence the formation and the amount of deposits. In the MCRT-100, one has only static test conditions, the condensate of the evaporated lubricant is not returned to the vials, and one has no lubricant/metal contact, which is believed to be a significant influence.

The major process during a test with the MCRT-100 is volatilization of the lubricants. This may explain why 3 cSt lubricants, which have a higher evaporation loss than the 5 cSt lubricant, produce a lesser amount of residue compared to the 5 cSt lubricants under the test conditions evaluated. Substantial oxidation and thermal degradation also occur, which forms residue in the glass vials. The residue after complete evaporation consists mainly of deposits from the breakdown of the lubricants with traces of nonvolatile additives.

The MCRT demonstrated a significant separation of lubricants meeting the same specification regarding their coking tendency. One can differentiate between low, medium, and high coking lubricants using the MCRT. The amount of remaining residue is not only a function of volatilization, but also a function of the amount of additives added to the lubricants. The chain length of the basestocks (ester) used may also influence the amount of residue. A comparison of test data from different coking deposition tests show that the MCRT-100 and the static coker deposition tester have a good correlation.

The lubricants from different specifications were separated in the same order with these test devices. The reason for this is most probably due to the similar test conditions. The coke-forming tendencies as determined using the MCRT, the BRG DEP, and the WADC DEP tester showed different trends. One lubricant which had the highest amount of residue in the MCRT and had not passed the J57 engine test because of excessive deposits, had an average amount of deposits in the BRG DEP tests and the second lowest amount of coke deposits in the WADC DEP. The basis for the wide range of test results obtained from the different tests is attributed to the nature of the different test conditions. In the BRG DEP and the WADC DEP tests, one has dynamic conditions and the stressed oil constantly replenished during the tests. However, in the MCRT, one has static conditions and lubricants were not replaced during the tests. These differences reinforce the need to select a test which simulates a specific type of engine coking prior to its use as an evaluating technique. A combination of the above tests should be employed to screen candidate lubricants prior to a decision to pursue additional evaluation using expensive and time consuming engine simulator or engine tests.

2. RECOMMENDATIONS

a. Since the repeatability of the MCRT-100 was excellent and a differentiation between lubricants from the same specification is possible, the MCRT-100 should be used as one tester in the MIL-L-7808J, the MIL-L-23699C, and the D.ENG.RN 2497 specification.

b. Some tests with the special 1/2 dram vials (7.2 cm instead of 3.2 cm) should be conducted to determine if improved ranking of lubricants is possible.

c. Correlation tests with different laboratories should be performed.

d. Tests with different base oils, full formulations and full formulations plus a increased amount of additives should be performed to see how the concentration of additives in lubricants affect the amount of remaining residue in the glass vials.

REFERENCES

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2. Military Specification, MIL-L-23699C Lubricating Oil, Aircraft Turbine, Synthetic Base (June 1981).

3. Dr E. Jantzen, "Formation of Deposits in Aircraft Engines," Deutsche Forchungs- and Versuchsanstalt Fuer Luft- and Raumfahrt E. V. Institut Fuer Technische Physik Stuttgart.

4. Deposition Test (WADC) according to Military Specification MIL-L-7808J Lubricating Oil, Aircraft Turbine Engine, Synthetic Base (May 1982).

5. Bearing Deposition Test in accordance with the test method defined in Appendix 1A of MIL-L-27502 and MIL-L-7808J.

6. Military Specification, D.ENG.RD 2497 Lubricating Oil, Aircraft Turbine Engine, Synthetic Base.

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